MIRL REPORT NO. 79

USING POLYETHYLENE AS A COAGULANT FOR REDUCING TURBIDITY FROM PLACER MINING DISCHARGE

A

Thesis

Presented to the Faculty of the
University of Alaska in Partial Fulfillment
of the Requirements
for the Degree of

MASTER OF SCIENCE

By

Ray-Her Fan, B.S.

Fairbanks, Alaska

September 1987

Library of Congress Cataloging in Publication Data

Library of Congress Catalog Card Number 88-060570
ISBN 0-911043-06-3

September, 1987

Published by

Mineral Industry Research Laboratory
210 O'Neill Research Laboratory
University of Alaska Fairbanks
Fairbanks, Alaska 99775-1180

ABSTRACT

Placer gold mining locations on Gilmore and Crooked Creeks in the Fairbanks and Central/Circle, Alaska areas, respectively, were chosen as study sites for evaluation of a unique water treatment process.

The physical and chemical impacts on water quality by placer mining were investigated by measuring the pH value, turbidity, and solids content of the slurry samples. Sedimentation tests, zeta potential measurements, and particle size distribution analyses were conducted as well. Also analyzed were mineralogical and chemical composition of the suspended ultrafine particles.

Flocculation tests using polyethylene oxide (PEO) with adjunct additives were conducted in the laboratory. Variable parameters such as mixing speed and time, reagent dosages, pH values, as well as synergistic factors were studied. Economic factors and chemical consumption were evaluated and a field treatment plant was designed and proposed.

ACKNOWLEDGMENTS

I want to express my deepest appreciation to my committee chairman, Dr. David R. Maneval for his guidance and instruction in the conducting of this project. A sincere acknowledgment is offered to Dr. Donald J. Cook and Dr. Timothy Tilsworth, committee members, for their help in sample collection and reviewing this work.

I am grateful to the staff of the Mineral Industry Research Laboratory for their help in the operation of some analytical equipment.

Finally, I would like to acknowledge financial assistance from the U.S. Bureau of Mines. This research has been supported by the Department of the Interior's Mineral Institutes program administered by the Bureau of Mines under allotment grant number G1164102. Without its support, this project could not have been accomplished.

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CHAPTER I

INTRODUCTION TO THE PLACER MINING INDUSTRY

Placer Mining History

The mining of placer gold can be traced back to 1790 when initial mining of gold occurred near Sitka. After that, some low-grade gold placers or lodes were discovered in Juneau, near the Yukon River, and along the Seward River. The discovery of gold near the Forty Mile River in 1886 was the first breakthrough to the interior of Alaska. By 1895, the new town of Circle had a greater population than that of the Forty Mile (Cook, 1983).

Placer gold production reached peaks in the 1910's and again in the 1940's (Wolff, 1982). The discovery of gold in the Seward Peninsula, the Tanana River, and the upper Koyukuk River was the major reason for the peak in 1910. The technological advances in mining method and the price of gold stimulated the production; thus, a peak of 800,000 oz was obtained in 1940 (Cook, 1983; Bundtzen, et al., 1985).

Like all metals, the production of gold in Alaska was influenced by discoveries of new mines, fluctuations of gold price, technology development and political events. Among those, new discoveries and the establishment of mining districts had the most obvious effect.

The decline in production after 1910 was due primarily to no new discoveries, depletion of reserves and the advent of World War I. From the 1930's on, a rapid increase was noted over the next 10 years to the highest production peak in 1940. Contributing factors were large increases of mining activities and the boost in the price of gold. After the peak year of 1940, the production declined sharply due to War Production Board Order L-208. In 1944, production was only 50,000 oz, the lowest point since the year 1900 (Cook, 1983; Bundtzen, et al., 1985).

The gradual lifting of the War Production Board ban raised production. A gradual decline occurring from 1944 to 1970 was a result of increase of production costs and the fixed price of gold. Finally, the recovery of gold prices in the early 1970's had attracted more people into this industry. As a result, the annual production increased to 190,000 oz by 1980. The production and value of placer gold from 1900 to 1985 are shown in Figure 1 and Figure 2 respectively.

Placer Mining's Economic Role

The placer mining industry is currently playing a important role in the economic development of Alaska. By using input/output analysis, Louis Berger and Associates (1983) conducted a study and estimated that placer mining contributed from 230 to 240 million dollars, 4.3% of the 1983 gross personal income for the State in 1982. It also created a total of 2940 jobs for the market. In 1983, there were about 296 commercial placer mines and about 40 recreational ventures which produced a total of 169,000 oz of gold and 18,000 oz of by-product silver. Within the error limits, gold production remained almost the same as 1982. Similarly, the average annual price of gold during 1983 was about the same as the previous year (Bundtzen, et al., 1983).

About 250 mechanized placer mines and 25 to 30 recreational-mining ventures produced at least 175,000 oz of gold and nearly 20,000 oz of silver in 1984. The 12% drop in the average annual price was probably the major reason for the decline in the number of mechanized operations. The value of the produced gold and silver was about $63,000,000 and $159,000 respectively (Bundtzen, et al., 1984).

An estimated 188,500 oz of gold and 27,400 oz of silver were produced by about 266 placer mines in 1985. The gold production increased even though the number of mines decreased from 281 to 266. The entire increase was from four large mines which contributed 34% of the annual production. The production from other small operators decreased slightly due to difficulties in low metal prices, water shortages, and exhaustion of reserves (Bundtzen, et al., 1985).

Peterson and others (cited in Bundtzen, et al., 1985) concluded that a total of 2,226 miners was employed in the whole industry, including those involved in recreational and assessment activities. An estimated $63.4 million were expended by the placer industry within the state, and the total impact of these expenditures on sales and income was 127.4 million dollars.

In general, the production of placer gold remained above 150,000 oz in the last few years, and the value remained about $60,000,000. Even though the placer mining industry is continuing to deal with such difficulties as wastewater clarification problems, exhaustion effects, low gold prices and water shortages, the placer mining industry will still be significant for Alaska's economy in the future.

CHAPTER II

WASTEWATER PROBLEM IN PLACER MINING

Legislation and Regulations

For more than a century, placer mining operations in Alaska have utilized water for the purpose of stripping, thawing, and sluicing for gold recovery. The privilege of

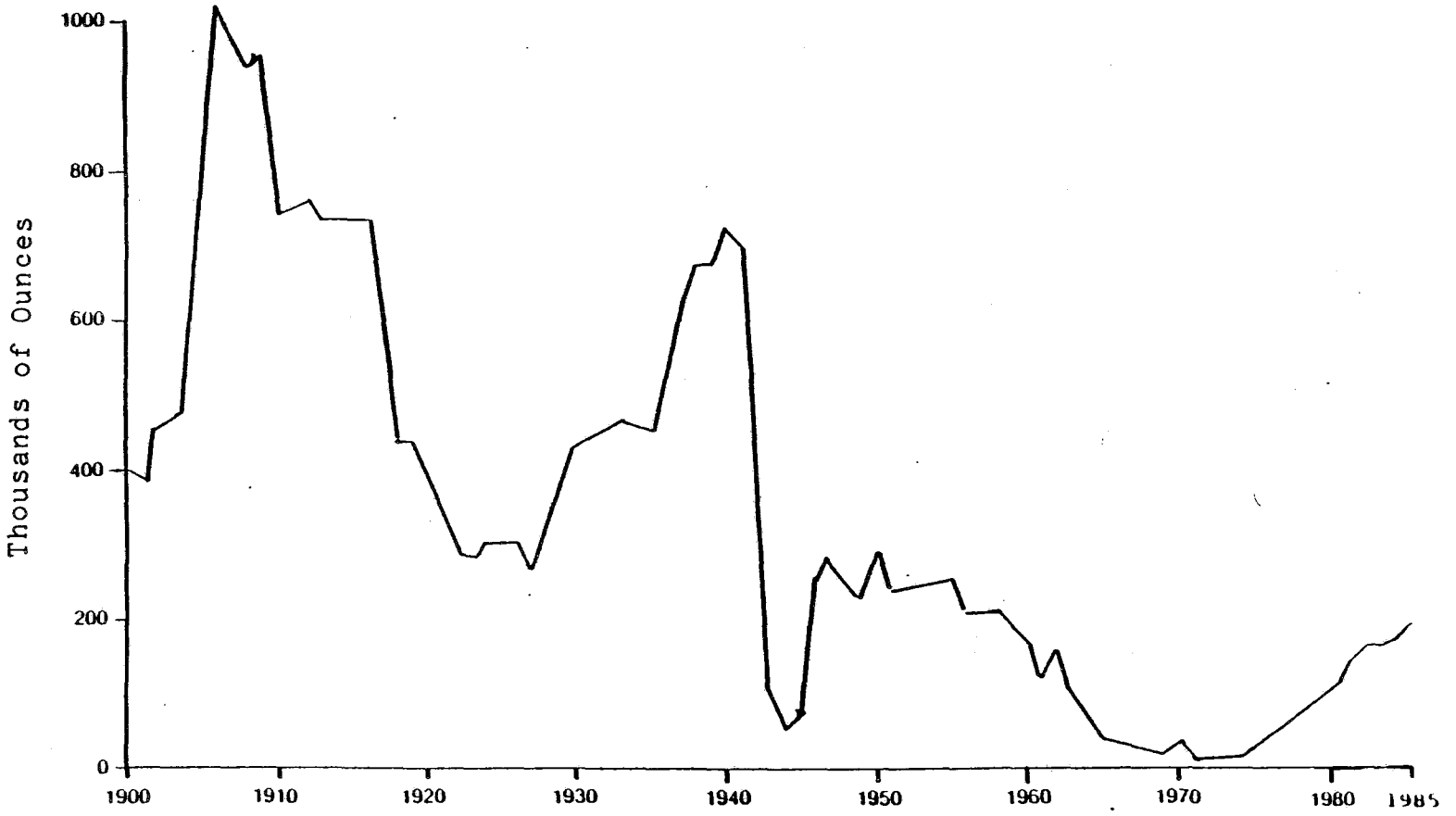


Figure 1. Placer gold production in Alaska, 1900 to 1985 (cited from Cook, 1983).

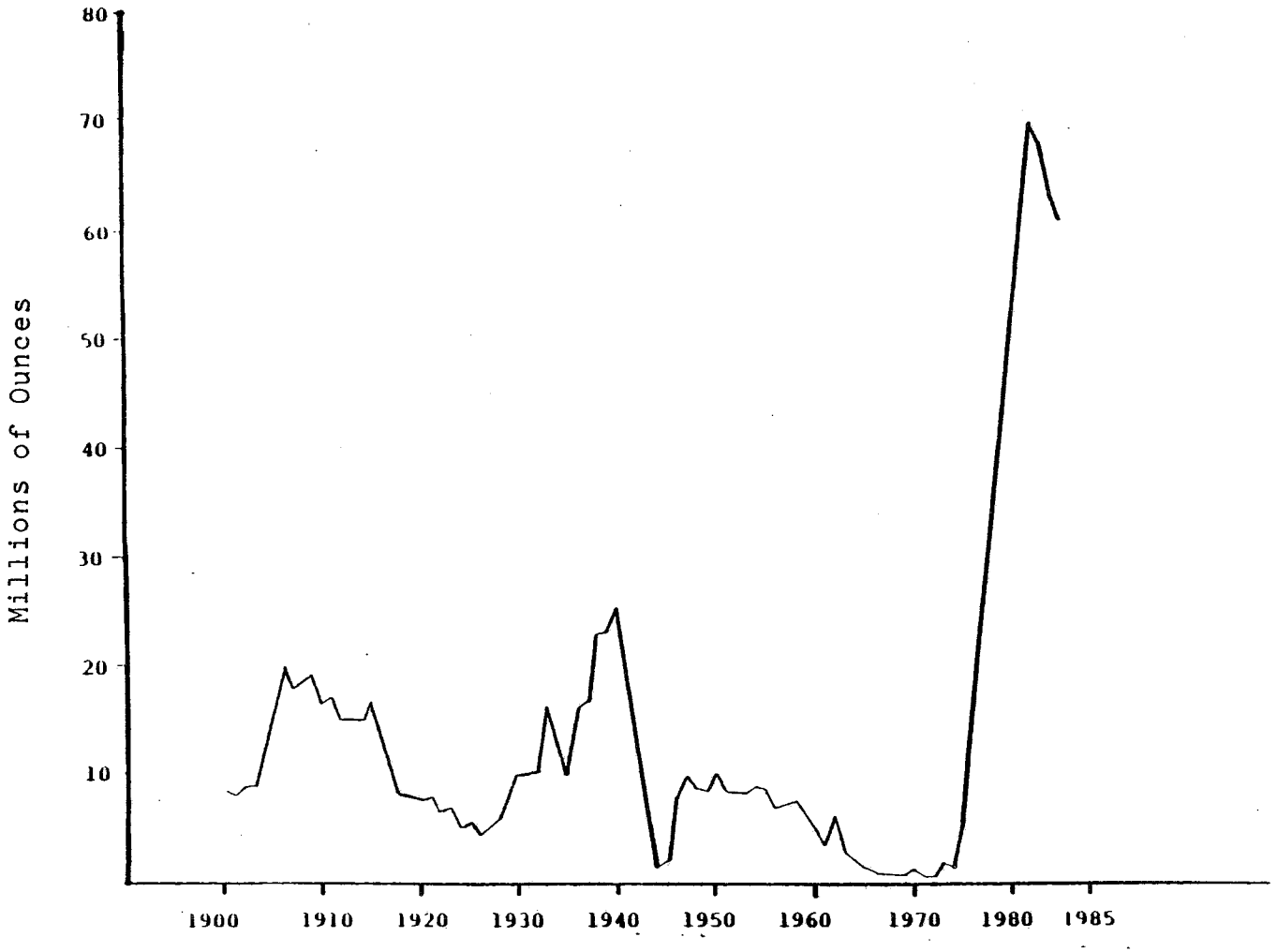


Figure 2. Placer gold production in terms of dollars in Alaska, 1900 to 1985 (cited from Cook, 1983).

unlimited water usage was changed as a result of the passing of the Water Pollution Control Act Amendment by Congress in 1972. The interim and permanent guidelines implemented by the Environmental Protection Agency (EPA) had significant effects on placer mining in Alaska.

The Water Pollution Act Amendment became Public Law 92-500 on October 18, 1972. The objective of this act is to "restore and maintain the chemical, physical, and biological integrity of the Nation's water" (U.S. Congress, 1972). As a result of this Act, both Federal and State agencies were responsible for establishing guidelines to achieve the goal of eliminating the discharge of pollutants into navigable waters by 1985.

In order to meet this Act, the following requirements were established:

(1) The application of the best practicable control technology currently available (BPT) should be established by July 1, 1977.

(2) The application of the best available technology economically achievable (BAT) should be established by July 1, 1983.

Interim effluent guidelines for all types of mining, with the exception of coal, were published by the EPA in 1975. This was followed by the Act's first mechanism, the National Pollution Discharge Elimination System (NPDES), to assure the compliance with the requirements of the Act. The State of Alaska was given the choice of administering the NPDES program or establishing its own. The alternative chosen resulted in a system by which EPA regulations are administered by the newly formed Alaska Department of Environmental Conservation (ADEC). In 1976, the EPA issued the first NPDES criteria to substitute the interim effluent limitation. Accordingly, the requirements for all water discharges from placer mining were limited to:

(1) The permittee will take reasonable steps to reduce the amount of organic and nonorganic solids discharged to navigable water.

(2) The permittee shall prove safe passage of fish around or through the mining area.

(3) The turbidity caused by the permittee should not be greater than 25 Jackson Turbidity Units (JTU) above the receiving water level. This turbidity is to be measured at a point 500 feet downstream from the final discharge.

(4) One of the following conditions must be met by permittee:

(a) The volume of settling ponds should be capable of containing the water used in any day's operation. This alternative requires no monitoring.

(b) The maximum daily concentration of settleable solids from a mining operation shall be less than 0.2 ml of solids per liter of effluent. This alternative requires monthly sampling with a monthly report by the permittee (Chang, 1979).

Under the Federal Water Pollution Act Amendment, Alaska Water Quality Standards were established as Title 18, Chapter 70 of the Alaska Administration Code in October 1973 and amended in 1979. These standards were essentially the same as those of the EPA and the NPDES permits required certification by the ADEC. The key criteria for the Water Quality Standards are:

(1) Dissolved oxygen shall be greater than 7 mg/L in water used by anadromous and resident fish.

(2) pH value should not be less than 5.0 or greater than 9.0.

(3) Turbidity shall not exceed 25 NTU (Nephelometric Turbidity Units) above natural condition level.

(4) Temperature shall not exceed 20°C at any time.

(5) No imposed loads of sediment will be allowed that will interfere with established water supply treatment levels (Chang, 1979).

In addition to the Alaska Department of Environmental Conservation (ADEC), the Alaska Department of Fish and Game (ADFG), and Alaska Department of Natural Resources (ADNR) are other state agencies that are concerned with the quality of mining discharges. But it is the ADEC's responsibility to promulgate and enforce regulations to prevent water pollution.

Although the EPA established final BPT and BAT criteria by regulation, it still reserved the limitations for gold placer mines. It was felt that the technological data available for placer mining was severely lacking and an ongoing survey of placer mining was initiated. A new date of July 1, 1984 was established for all industries to meet BPT for conventional pollutants and BAT for toxic pollutants. A list of Placer Mining Technology Standards proposed by EPA in April 1985 is included in Table 1. But with the uneventful passing of these dates, it appears that 1987 will be the deadline to meet these regulations (Lipchak, 1986).

Table 1: EPA proposed Placer Mining Technology Standards in April 1985

Mine Description	Ore Production yd^3/Day (24 hr)	Treatment Required	Water Standard	Treatment Level
Recreational & Assessment	20	None	None	None
Any Surface Placer Mine	20-500	Settling Pond	1. Settleable solids Daily max = 1 ml/L 30 day average = 0.3 ml/L 2. Total suspended solids 30 day average = 2000 ppm	BPT
Any surface Placer Mine	More than 500	Settling Ponds and 1. 80% recycle and/or 2. Flocculants and/or 3. No discharge (100% recycle)	0 TSS	BCT & BAT
Large Dredges	More than 4000	No discharge (100% recycle)	0 TSS	BCT and BAT

* As levels to be set later
Hg levels to be set later

**
Mine Drainage: Seepage water volume must equal BPT quality
Storm relief: 6 hr. retention over 5 yr/6 hr. storm rainfall event is "free", (1.25" in Fairbanks) freeboard
Site runoff: diversions expected

At a public meeting held in Fairbanks on March 28, 1987, new proposed effluent limitations were announced by EPA. This material is summarized in Appendix A and would be known as 40 CFR Part 440.

Mining Methods

The methods used by placer mining in most areas can be divided into two systems:

(1) Hydraulic Methods— Stripping overburden, moving gold-bearing gravel to the sluice box and stacking tailings processes are accomplished by hydraulic giants.

(2) Mechanical Methods— Using a bulldozer and other heavy equipment, the valuable gravel is moved to a sluice plate ahead of the sluice box. The pay gravel is then washed through a sluice box to recover gold. The overburden and valueless tailings are also moved to a disposal area by mechanical equipment.

Large quantities of water are consumed in placer mining, even in the mechanical method. Thus, it introduces large quantities of solids into the water, and creates water quality problems. In this project, the slurry samples from Gilmore and Crooked Creek areas were studied for possible water clarification.

1. Gilmore Creek Area

The mechanical method is utilized in this area during the mining season (from June to the beginning of October). Three bulldozers are used to strip overburden, move gravel and stack tailings. After the gravel is moved to the sluice box, a hydraulic giant is used to break it up and wash it down to the sluice box. The tailings are moved away to the side of the processing plant for disposal.

There are five settling ponds placed downstream of the sluice box along the valley. The distance between each pond ranges from 50 to 150 feet. Each pond is estimated to be at least one third of an acre in area. The slurry from the sluice box flows into the end of the first settling pond. Some water is pumped as recycle water from the side. Underground water seepage, a small flow of freshwater and surplus muddy water from the first pond serve as the water supply of the second settling pond, and so on. Overflow from the last pond flows into the Gilmore Creek.

2. Crooked Creek Area

In this area, the operating method is same as that at the Gilmore Creek area. There are several bulldozers utilized for stripping overburden and transporting gravel as well as tailings. There is only one settling pond for containing and recycling operating water. The water coming from the sluice box flows into the first pond via a ditch, and is pumped up by steel pipes to wash gravel on sluice plate. Additional ponds will be constructed when the mining operation is moved upstream.

To assure sufficient settling, it is advisable that the length of a settling pond should be at least twice as long as its width. If not, baffles such as wood or plastic barriers, or tailings should be placed in the pond to increase the flow path of the water, thus preventing short circuiting. For both mining techniques mentioned above, there were no such energy dissipation devices employed.

Scope and Objective

In order to meet the Federal and State water quality regulations, some research projects are being conducted under a grant of the U.S. Bureau of Mines. According to Zemansky, et al. (1976), the impacts from placer mining can be classified into three categories:

(1) Physical impacts such as settleable solids and suspended solids which result in turbidity.

(2) Chemical impacts such as decomposition of organics that will decrease the dissolved oxygen level in the water and change the pH value.

(3) Physical and chemical impacts that will cause biological effects on aquatic life.

In this project, the following variables will be studied:

(1) General characteristics of slurry samples such as pH value, specific gravity, turbidity and solids content will be measured.

(2) Analysis of physical properties such as sedimentation tests and particle size analysis will be conducted.

(3) Mineralogical and chemical composition of the fine particles will be analyzed.

(4) Coagulation and flocculation treatment will be carefully accomplished.

By means of laboratory experiments and a literature survey, the following objectives will be sought:

(1) The determination of effect of placer mining on water quality.

(2) The effectiveness of flocculation treatment by the chemical reagent, polyethylene oxide.

(3) Practical and economic design of wastewater treatment plants.

CHAPTER III

TREATMENT BACKGROUND AND THEORY

Placer mining in Alaska has been required not only to remove silt size and settleable solids but to reduce the suspended solids resulting from their mining operation. For silt and settleable solids, well designed settling ponds will accomplish the clarification work. The real problem for placer miners is the ultrafine particles that are suspended for a long period of time unless special treatment is utilized.

Before the study of fine particles, it will be useful to define the terms silt, clay and colloid. In most mechanical analysis of soil, silt has a grain size between 0.002 to 0.06 mm (below 44 mesh to about 230 mesh). Sand is considered to have larger size than silt. Clays are considered to be particles that are less than 0.002 mm in diameter (Maneval, 1985). Clay, as used in soil science, does not have to mean the mineralogical material known as clay, but is a description of materials that have a particle size as mentioned. Particles that are less than 0.0001 mm are referred to as colloids.

Most Interior Alaska placer mines and mines in the nearby Yukon likely consist of a high percentage of ultrafine materials such as colloid and clays particles (Chang, 1979; Yang, 1979).

Characteristics of Fine Particles

An analysis of fine components from Interior Alaska placer mines was conducted by Maneval (1985). These samples were analyzed semiquantitatively by x-ray diffraction techniques. All fine materials was found to contain illite, and several were found to contain kaolinite. Chlorite also was found in some of the samples. Accordingly, most of the "clays" in placer pay dirt are clay minerals indeed.

The settling rates of particles in a quiescent fluid are governed by Stokes' Laws. They are dependent on the particle size, particle density and liquid density. The equation of Stokes' Law for spherical particles is as follows:

$$\text{Stokes' Law:} \quad V = \frac{2gr^2(d_1-d_2)}{9v}$$

where V is the final velocity of the sphere, r is the radius of the sphere, d_1 is the density of the sphere, d_2 is the density of the liquid, v is the coefficient of viscosity and g is the gravitational constant. Table 2 shows the calculated times for a particle with a density of 2.0 g/cc to settle one foot as the diameter of the particle decreases. The table also shows how the total surface area increases as the particle size decreases. From Table 2, the difficulty of fine particles to settle out is obvious (Connelly and Richardson, 1984).

TABLE 2. Effect of particle size on settling rate. (cited from Connelly and Richardson, 1984)

Diameter of Particle, mm	Order of Size	Area*, cm	Total Surface Settle 1 ft**	Time Required to
10	Gravel	3.142		0.03 sec.
1	Coarse sand	3.142 x 10		0.3 sec.
0.1	Fine sand	3.142×10^2		33 sec.
0.01	Silt	3.142×10^3		55 min.
0.001	Clay	3.142×10^4		92 hr.
0.0001	Colloid	3.142×10^5		384 days
0.00001	Colloid	3.142×10^6		105 years

* Area for particles as indicated size produced from a particle 10 mm in diameter.

** Calculation based on a sphere with a specific gravity of 2.0 at a temperature of 25°C.

Besides Stokes' Law, electrostatic charge on the particles can also strongly influence their behavior in settlement. Agglomeration of particles will result in a faster settling rate, but occurs only if there are collisions and attachment between particles. Collisions will take place due to thermal (Brownian) motion and also will be enhanced while inducing velocity in the slurry. But, the formation of agglomerate mostly depends on the balance of attractive and repulsive forces between the particles. These two forces will be discussed in detail in the following sections.

Origin of Colloid Stability

It has been observed that solid particles in a colloidal dispersion (the solid and the aqueous phases together) can move in an electric field. This fact indicates that these particles carry electric charges. Most colloidal particles develop a negative charge in wastewater or water. It has been noted that a colloid dispersion has not any net electrical charge. As a result, the primary charge on the particle surface must be balanced in the aqueous phase. Therefore, the charged particle and opposite charged ions (counter ions) diffusing in dispersions will form so-called electric "double layer".

Repulsive forces will arise between colloidal particles due to these double layers. These repulsive forces contribute significantly to the stability of the colloidal particles. It is therefore useful to consider the origins of the primary

charges which are responsible for these repulsive interactions. There are three different processes able to bring about the primary charge.

First, a surface charge may arise from imperfections within the crystal lattice of particles (isomorphic replacement). The primary charge of clay minerals belongs to this category. The principal building elements of the clay minerals are two-dimensional arrays of silicon-oxygen tetrahedra and two-dimensional arrays of aluminum- or magnesium-oxygen-hydroxyl octahedra. The basic structure of clay elements are shown in Figure 3.

In most clay minerals such sheets of tetrahedra and of octahedra are superimposed in different fashions. In the tetrahedral sheets, tetravalent Si is sometimes partly replaced by trivalent Al. In the octahedral sheets, there may be replacement of trivalent Al by divalent Mg without complete filling of the third vacant octahedral position. Al atoms may be replaced by Fe, Cr, Zn, Li, and other atoms. The small size of these atoms permits them to take the place of the small Si and Al atoms. Since an atom of lower positive valence replaces one of higher valence, a deficit of positive charge, or in other words, an excess of negative charge will be generated (Van Olphen, 1977).

Second, the primary charge on colloidal particles may arise because of ionization of chemical groups present at the surface of the particles. For instance, proteins which contain free carboxyl (-COOH) and amino ($-NH_2$) groups will carry a positive charge at pH values about 4 ($-COOH + H_2O = COO^- + H_3O^+$), and a negative charge at pH values below 10 ($-NH_2 + H_3O^+ = -NH_3^+ + H_2O$). The charge on these particles is then dependent on the extent to which these particle surfaces ionize; thus, particle charge depends on the pH of the solution (O'Melia, 1969).

Third, the surface charge on the colloid may arise from a preferential adsorption of certain ions from solution. These "potential determining ions" are often identical with those constituting the crystallite. For example, Ag^+ and I^- are specially adsorbed on the surface of a AgI dispersion. The specific adsorption of ions may arise from hydrogen bonding, covalent bonding, or van der Waals bonding (Van Olphen, 1977). Note that when the primary charge arises from either ionization of surface group or preferential adsorption of certain ions, the sign and magnitude of the charge depends largely upon the characteristic of the aqueous phase.

The primary charge on the colloidal particles brings about a repulsive force when particles are brought near together, and thereby tends to prevent particle aggregation. These repulsive interactions may be overcome, i.e., the mechanisms by which colloidal particles are destabilized.

Colloid Destabilization

Aggregation of colloidal particles is the prime objective of destabilization processes. This aggregation can be achieved in several different ways. Polymeric chemicals can function in a completely different manner compared with traditional coagulants such as aluminum (III) and iron (III). Over the last several decades, utilization of synthetic organic polymers in solid/liquid separation processes has increased dramatically. Therefore, emphasis will be placed on synthetic polymers instead of traditional coagulants in this study.

A number of mechanisms are able to explain colloid destabilization. Two models which seem to have theoretical and practical significance will be summarized here; a) the electric double layer model and b) the chemical bridging model (O'Melia, 1972).

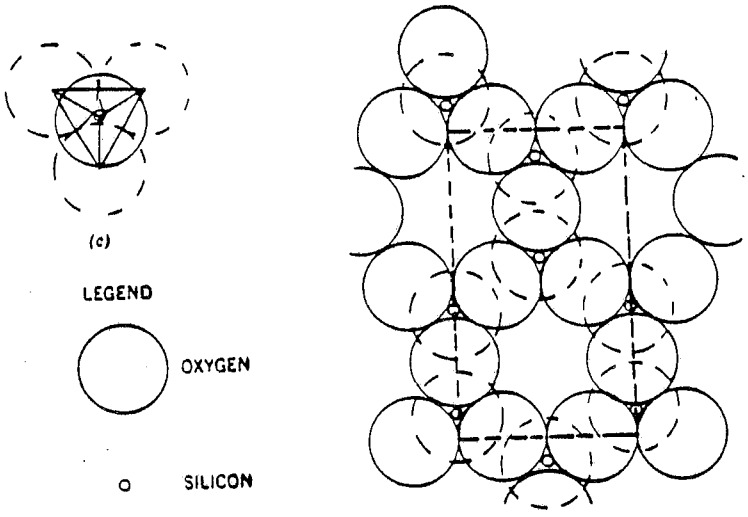
1. Electric Double Layer Model

The electric double layer is the simplest colloid-chemical model that can be used to describe the interactions among colloidal particles. First one considers the distribution of ions in the diffuse layer. Counter ions are attracted to the oppositely charged surface and give rise to a concentration gradient, where these ions diffuse away from the surface towards the bulk of the solution. In the diffuse layer, the concentration of counter ions is highest adjacent to the particle surface and decreases gradually with increasing distance from the solid-liquid interface. Such a distribution is shown schematically in Figure 4-A.

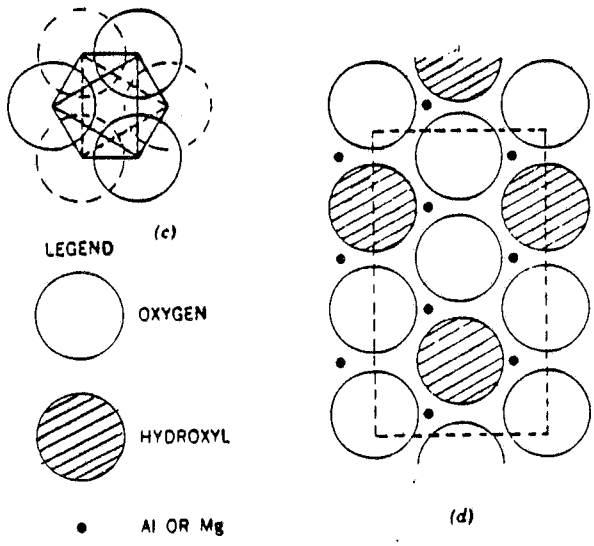
A quantitative model for the diffuse layer which permits calculation of the distribution of both the counter ions and ions of the same sign as the particle charge was developed by Gouy and Chapmen in about 1910 (O'Melia, 1972). From the Gouy-Chapmen model, it is possible to calculate the electric potential (voltage) at any point in the diffuse layer with respect to the bulk solution. A schematic representation of the potential distribution is shown in Figure 4-B. The potential has a maximum value at the particle's surface and decreases approximately exponentially with distance from a particle's surface (O'Melia, 1969).

When two similar colloidal particles approach each other, their diffuse ions atmospheres begin to interact. It can be shown that the interaction between these particles of similar primary charge leads to a repulsion between the particles. A repulsive potential energy (V_R) may be determined and it varies approximately inversely in an exponential manner with the separating distance. Such an interaction is shown in Figure 4-C and 4-D (O'Melia, 1969).

Colloids can be aggregated by attractive forces existing between the particles. The electric double layer theory invokes van der Waals attractive forces between the atoms of the colloidal particles. These van der Waals forces are



Tedrahedral sheets



Octahedral sheets

Figure 3. Structure of the tedrahedral sheets and octahedral sheets (from Van Olphen, 1976).

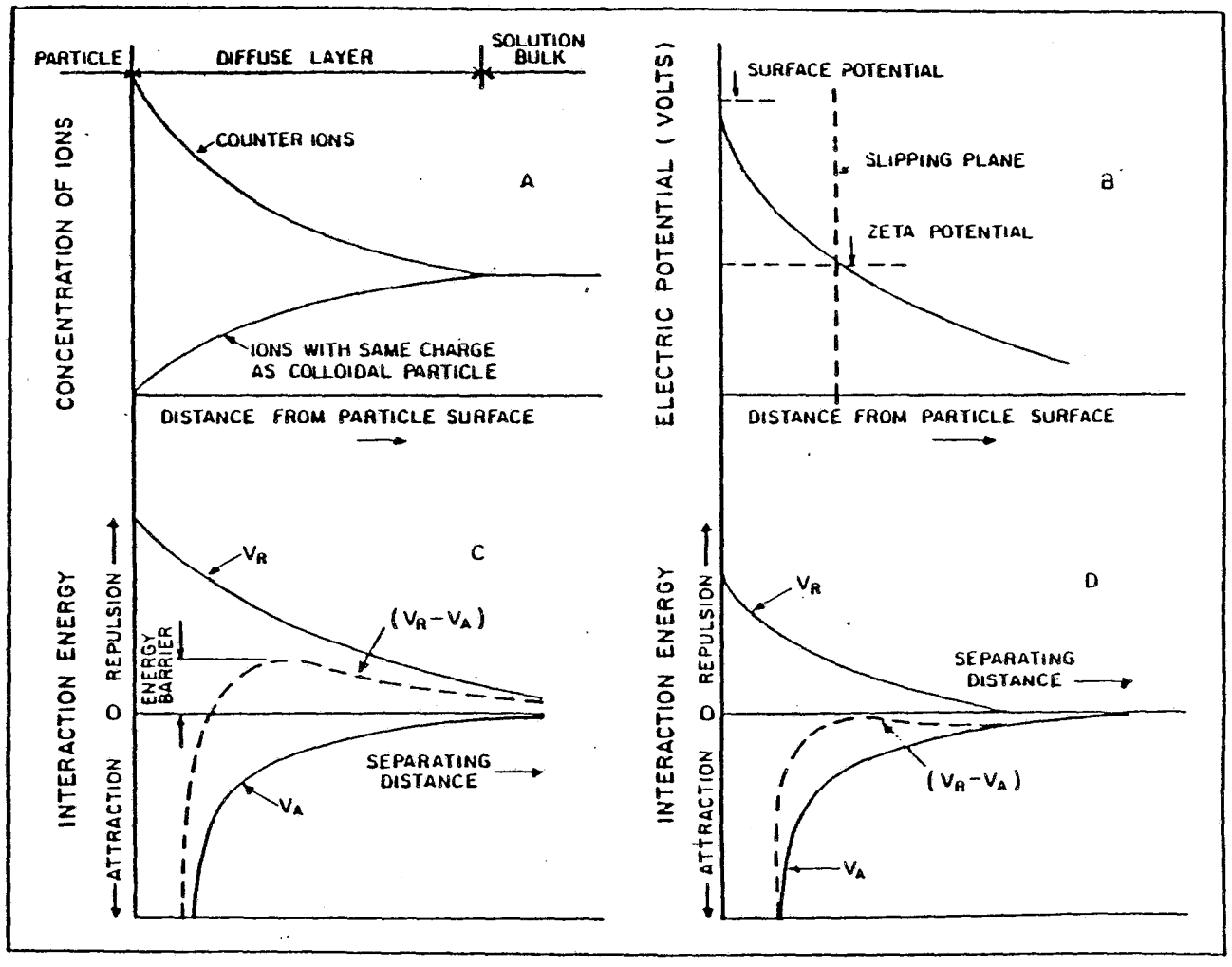


Figure 4. Schematic illustrations of the electric double layer model interactions for colloid stability (from O'Melia, 1969).

due to interactions between dipoles, either permanent or induced, and are always attractive. It depends on the kinds of atoms in the colloidal particles and on the density of the particles. Figures 4-C and 4-D also schematically show the van der Waals attractive energy of interaction (V_A) (O'Melia, 1969).

The van der Waals attractive potential energy is virtually independent of the composition of the aqueous phase, while the repulsive potential energy may be controlled by characteristics of both the aqueous and solid phases. Increasing the electrolyte concentration will compress the diffuse layer, and decrease the magnitude of the repulsive energy of interaction. Thus, it will eliminate the potential energy barrier, which is shown in Figure 4-D, and thereby destabilize the colloid (Van Olphen, 1977).

2. Chemical Bridging Model

It is essential to recognize that particle destabilization by some materials can not be characterized by the double layer model. The chemical bridging theory provides a more acceptable model for understanding the ability of polyelectrolytes to destabilize colloidal dispersions.

The chemical bridging model proposes that a polymer molecule can attach itself to the surface of the colloidal particle at one or more adsorption sites, with the remainder of the molecule extending into the solution (Figure 5-A). These extended segments can then interact with vacant adsorption sites on another colloidal particle, forming a chemical bridging between the particles. This action is shown in Figure 5-B. After the suspended particles are bridged, flocs are generated and able to settle rapidly. Optimum destabilization occurs when only a fraction of the available adsorption sites on the surface of the colloidal particles are covered.

Absence of appropriate mixing and overdose of chemicals which saturate the available surfaces of the dispersed phase will produce a restabilized colloid, because no sites are available for the formation of polymer bridges. This situation can be shown in Figure 5-C and 5-D respectively. Excess shear forces will also cause restabilization owing to disruption of bridging bonds. Such actions are shown in Figures 5-E and 5-F (O'Melia, 1969).

Besides the two theories mentioned above, for clay particles, some other phenomena also have to be considered. In clay mineralogy, cations will be absorbed on the surface layer in order to compensate for the unbalanced charge in the interior of layers due to isomorphous replacement. The absorbed cations are exchangeable and can be determined analytically in terms of cation exchange capacity (CEC). Van Olphen (1977) indicated that naturally occurring Ca-clays are more unstable and easy to be flocculated in a dispersion system. This phenomenon was also confirmed by Stanley and Scheiner in 1983. In their ion-exchange research of the clay flocculating process, a conclusion was made that the efficiency of performance was inversely proportional to the ratio of ionic radius to charge (R/C).

Another phenomenon in clay peptization is that the edge of a clay particle will carry a positive charge in dispersion when the pH value is less than 7. The positive edge charge may be expected to be reversed upon the addition of alkali. Therefore, keeping clay solution in an acid condition will increase the formation of aggregates. The reason is that positive edges will be easily attracted by the negative charge of the clay surfaces (Van Olphen, 1977).

Coagulation and Flocculation

Chemical clarifiers such as coagulants and flocculants have been used in purifying water and wastewater for centuries. Such treatments also have been commonly used to treat mine effluent in the lower 48 states. In this section, general information of coagulation and flocculation will be reviewed.

Coagulation is often referred to as the process of reducing the surface charge of colloidal particles to the point where the attractive force can predominate and cause agglomeration of suspended particles. The chemicals which bring this about are referred to as coagulants. Flocculation is referred to as the process of agglomeration of suspended solids without changing surface charge substantially. The chemicals which perform such reaction are flocculants. Currently the most used coagulants and flocculants are organic and synthetic polymers.

A polymer is a molecule composed of many repeating units. A monomer is the building unit of a polymer. If a polymer has a positive charge associated with its backbone, it is referred to as being cationic. Conversely, if the polymer has a negative charge associated with its backbone, it is referred to as being anionic and if the polymer is uncharged, it is referred to nonionic polymer (Connelly and Richardson, 1984). Coagulants and flocculants are sold in various forms. Coagulants are normally sold in liquid form, while the flocculants are mostly sold as dry powder and emulsions (Dauplaise and Werneke, 1982).

Generally, coagulants have relative low molecular weights (200-2000 molecular units) in comparison to flocculants (5-20 molecular units). The polymerization of coagulants is a condensation process, which is sometimes referred to as step-growth polymerization, while the flocculants are made via addition polymerization. The molecular weight of both can be controlled and modified by changing the manufacturing processes, thereby both of them can meet specific applications. This is one of the reasons why they are popularly used in industry (Connelly and Richardson, 1984).

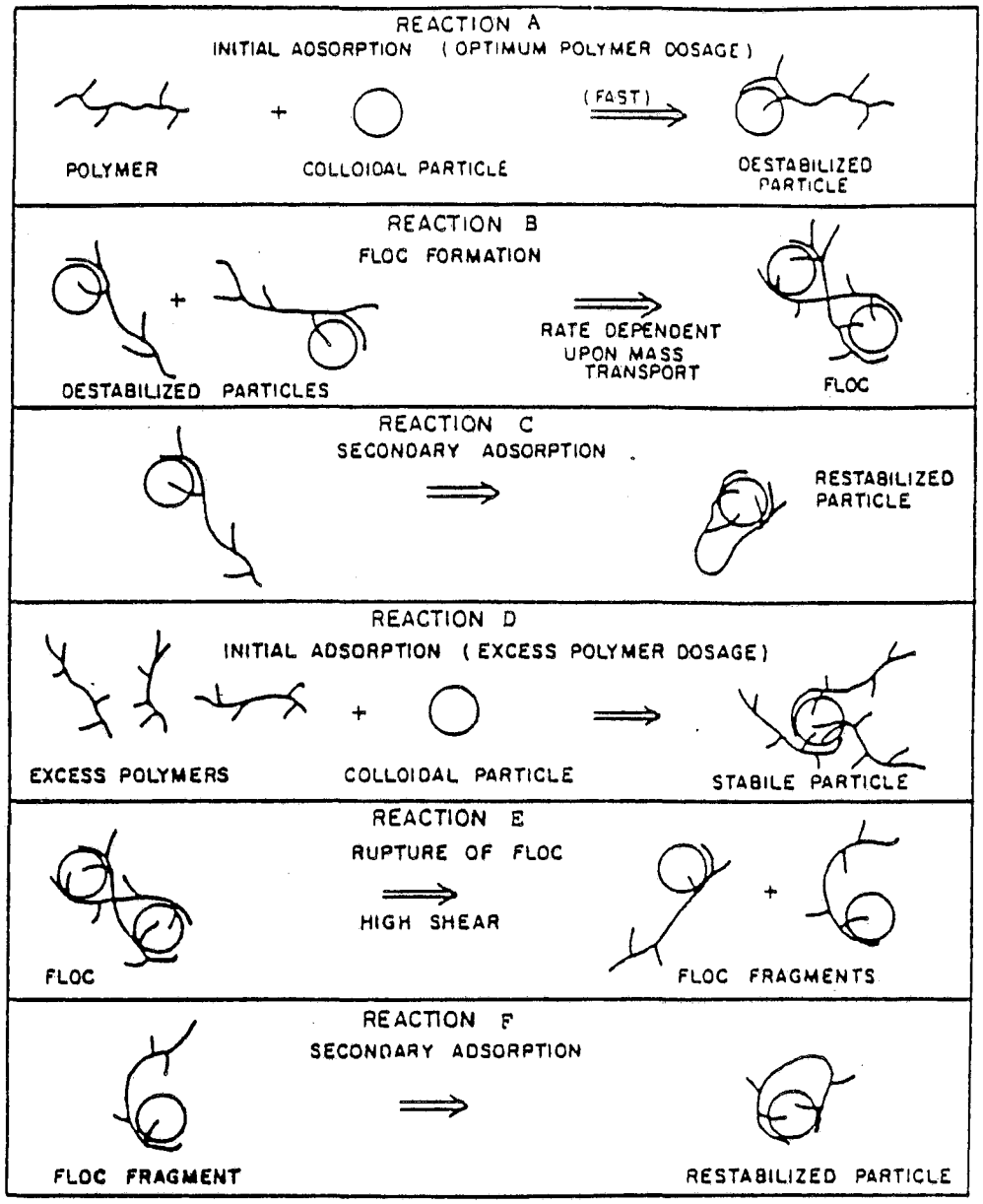


Figure 5. Reactions occur between colloidal particles and polymers (from O'Melia, 1969).

It has been concluded that cationic polymers serve a dual functions in the destabilization reaction. They serve as very effective reagents either by compressing the double layer or by bridging discrete particles. Anionic and nonionic polymers would not be expected to exhibit this dual function in the destabilization of negatively charged colloids (Birlner and Edzwald, 1969). By studying the adsorption of a polymer of ethylene oxide, Birlner and Edzwald (1969) indicated that polymer segments attached to the silica surface via hydrogen bonding. The mechanism has also been invoked by Howard and McConnel (cited in Birlner and Edzwald, 1969) to explain the adsorption reaction. It may be concluded that hydrogen bonding between polar groups plays a significant role in bridging model.

Polyethylene Oxide (PEO)

The Bureau of Mines, U.S. Department of the Interior over the past several years has been conducting research to reduce the adverse impact on the environment of waste slimes produced during the processing of minerals. It has already developed a technique for processing phosphate clay mine effluents (Smelley and Feld, 1979). This technique, using PEO as the flocculant, may possibly be applied to a wide variety of industrial waste (Smelley, Scheiner and Zatko, 1980). Accordingly, it is selected as principal flocculant in this study.

PEO is a nonionic and linear polymer with a very high molecular weight, 5 or 8 million. The chemical composition

of the polymer can be represented by:

$$(-CH_2—CH_2—O-)_n$$

where n refers to the number of monomer units in the polymer chain. The polymer is extremely soluble in water and many common organic solvents. The most important characteristic of PEO is that it does not have adverse environmental effects on the vicinity of the mining operation (Zatko, 1980). By using PEO as a flocculant, the goal of solving the placer mining wastewater problem will be sought.

CHAPTER IV

SAMPLE AND EXPERIMENTAL METHODS

Sampling Methods

The objective of sampling is to collect a portion of material for analysis, the result of which should accurately portray the natural properties of the sample. The sample must be representative of the original material, and therefore, the sample should be handled carefully to insure that no change in composition occurs.

Only a satisfactory sampling program will result in reliable analytical data. Unfortunately, no general stream sampling procedures have been established which could apply to all situations. The sampling procedure is always dependent upon the function, accuracy and detail desired in a water quality study. For general water sampling, the following suggestions should be taken into account in order to obtain a satisfactory sample (Black, Hayes H., 1952):

(1) The sample should be collected from where the water is homogeneous.

(2) When sampling sewers and deep narrow channels, a sampling point at two-thirds the water depth from the top is suggested.

(3) When sampling discharge through large conduits, the point of collection should be either rotated across the channel in wide channels or be determined by experiments in deep conduits.

(4) When sampling is done at open-channel conditions such as a creek, the sampling point should be selected where the velocity is sufficient to prevent deposition of solids. This point generally occurs near the center of the channel and two-tenths or three-tenths the depth below the water surface.

(5) The jugs or containers used for sampling should be clean enough to store the sample. A separate sampling container must be provided for sampling at each sampling point.

(6) The complete information including date and time of collection, origin of sample, and name of collection should be provided after each sampling.

In addition to sample collection and preservation, correct selection of sampling locations is also important for a reliable representative sample. Law (1975) suggested following considerations for water sampling:

(1) The sampling point should have maximum accessibility and safety for personnel.

(2) Insure that the site provides the information desired.

(3) Sampling should be at a point of maximum turbulence to avoid sampling from a stagnant area.

Water Sample

The types of liquid samples may be broadly classified into three groups: grab samples, composite samples, and integrated samples (Standard Methods, 1984). The type of samples provided for analysis is based on the purpose of the study. Most water analysis is performed on grab samples collected by plastic jugs or samplers.

The basic ideas for selecting the sampling stations are based on where the water quality has been drastically changed by man-made or natural factors, and where the discharged wastewater influxes into the stream. Therefore, the sampling sites should be located above and below major waste inputs such as sluice boxes and settling ponds. Attention also should be given to sites below the confluence of tributaries to examine the impact caused by wastewater.

For most placer mines in Alaska, settling ponds are the most popular technology used to treat process wastewater at the present time. In many cases it may be uneconomical and impractical to treat the wastewater entirely by the flocculation process. Thus, a combination of settling pond and chemical treatment will be a possibility which deserves to be studied. Therefore, in this study, sampling sites are located at the outlet of each pond to reveal the real functioning of the settling pond. A location about one mile away from the last outlet of the settling pond is also selected to study the impact on the vicinity.

Because of practical and economic difficulties of establishing an automatic sampling device, water samples were collected by hand in 5-gallon plastic jugs, and fully labelled.

Six water samples, namely A1, A2, A3, A4, A5 and A6 were obtained from Gilmore Creek area near Fairbanks, Alaska and two samples B1 and B2 from Crooked Creek area near Circle, Alaska.

The A1 sample was collected at the outlet of the settling pond which held the discharged water from the sluice box. The A2 sample represents overflow from the second settling pond about 200-foot downstream below the first pond. Samples A3, A4 and A5 were collected from the outlet of the third, forth and fifth settling ponds respectively. The effluent from the fifth pond flowed into Gilmore Creek where sample A6 was collected approximately 1.5 miles below the outlet point. The B1 sample represents inlet water from the only big settling pond which is connected to the processing plant by a 200-foot ditch. Sample B2 was collected at the opposite end of the location of sample B1. The study areas and sampling locations are shown in Figures 6 and 7 respectively.

Since sampling is not as easy as it appears, samples were carefully collected and handled in this study. All of the process, including precleaning containers, selecting location and collecting samples were careful done to obtain the best representative samples.

Experimental Methods

The purpose of this section is to describe the laboratory methods used by this study. The step-by-step procedures will be included in Appendix B and Appendix C. The measurements and tests conducted in this research consist of general and physiochemical characterization. Included also are mineralogical and chemical analysis of fine particles. Finally, the flocculation study procedures using PEO will be discussed in detail.

1. General Characteristics

A. pH Value Measurement

pH is a measure of the negative logarithm (base 10) of the hydrogen ion concentration in moles per liter. Pure water is balanced by H and OH ions, so that the pH of neutral water is 7. Water with a pH less than 7 is acidic and with pH greater than 7 is basic.

The pH value of water can be measured either colorimetrically or electrometrically. In this study, an electronic pH meter with a glass electrode was used to measure the pH of each water sample.

B. Solids Content and Specific Gravity Measurement

Several papers pertaining to flocculation studies indicated that slurries which have different solids content will result in different performances. For the purpose of uncovering the relation between solids content and flocculation efficiency, gravimetric solids content analysis was performed by an evaporation method.

Because of the small amount of suspended solids involved, a balance accurate to 0.0001 g was used. A drying oven was utilized to remove all moisture from a known quantity of the sample. A known weight of sample contained in a preweighed flask was evaporated to dryness and then weighted. The total solid weight can then be deduced and the solids content can be calculated as well.

C. Particle Size Distribution Analysis

Not many waters are absolutely free of suspended matter in the forms of turbidity and suspended solids. Some particles settle rapidly while others remain suspended for a long period of time. This phenomenon arises from different particle size, shape and density which are subjected to different gravity forces.

Wet sieving and pipette analysis are two gravimetric methods used to measure the size distribution of a water or solid sample. In this study, however, a Sedigraph (model 5000ET) particle size analyzer, produced by Micromeritics Corporation of Norcross, Georgia was used. The Sedigraph particle size analyzer measures the sedimentation rates of particles dispersed in a liquid and automatically interprets the data in accordance with Stokes' Law to yield a cumulative mass percent distribution in terms of equivalent spherical diameter.

The instrument determines, by means of a finely collimated beam of X-ray, the concentration of particles remaining at decreasing sedimentation depth as a function of time. The logarithm of the difference between initial (time = 0) and instantaneous (time = t) transmitted X-ray intensity is scaled and presented linearly as "cumulative mass percent" on the Y-axis of an X-Y recorder. Before the analysis is conducted, the slurry has to be dried, filtered, weighed and then mixed with Calgon solution (a dispersant) to obtain an appropriate sample (Micromeritics Instruction Manual, 1984).

2. Physiochemical Properties

A. Imhoff Cone Test

In order to determine the settleable solids volume and to examine the efficiency of settling ponds, each water sample was subjected to volumetric analysis with the Imhoff Cone. The procedure for this test is recommended in Standard Methods (1984) as follows:

> Fill an Imhoff cone to the liter mark with a thoroughly mixed sample. Settle for 45 minutes, gently stir the sides of the cone with a rod or by spinning, and then settle 15

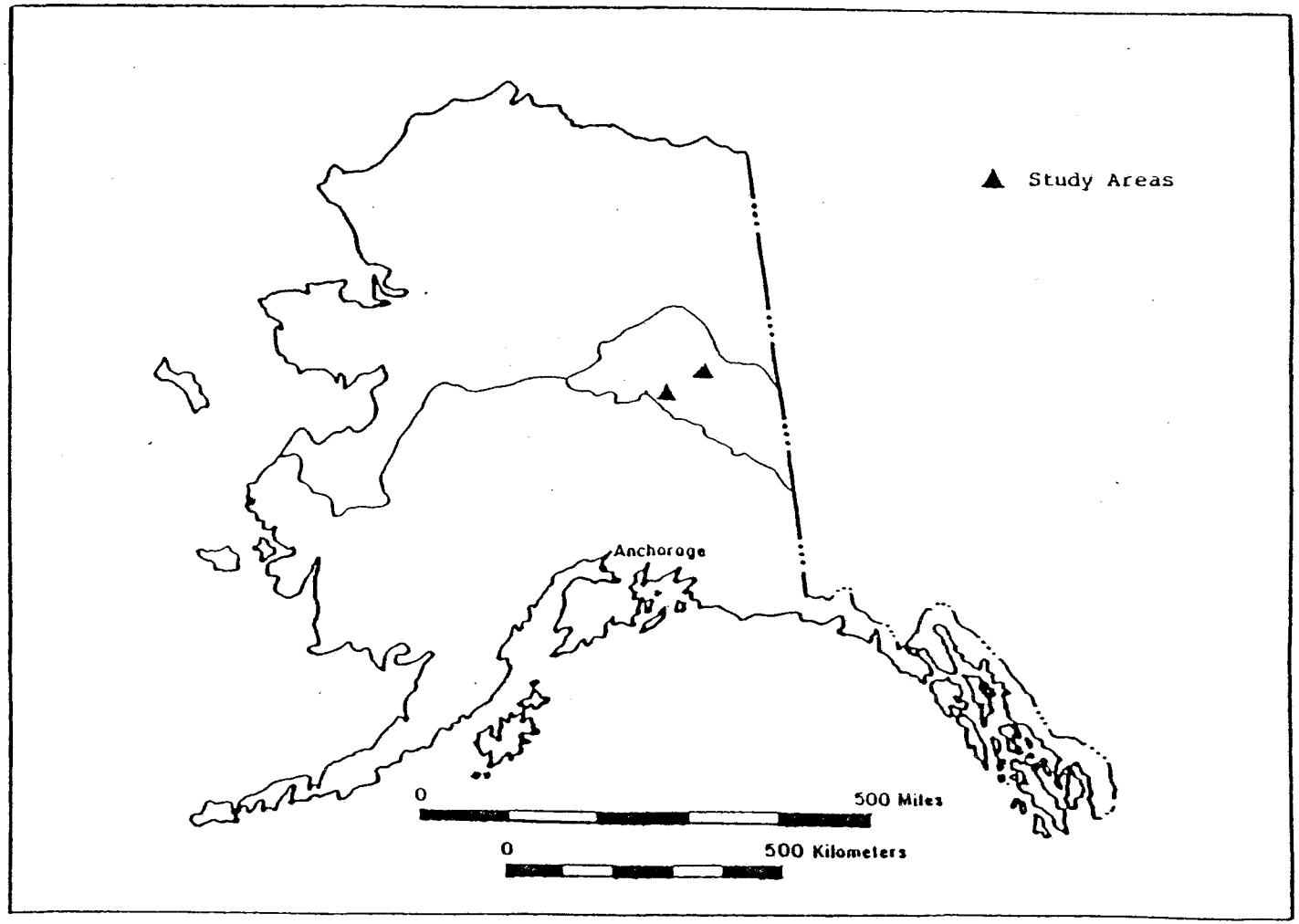


Figure 6. Study areas of this project.

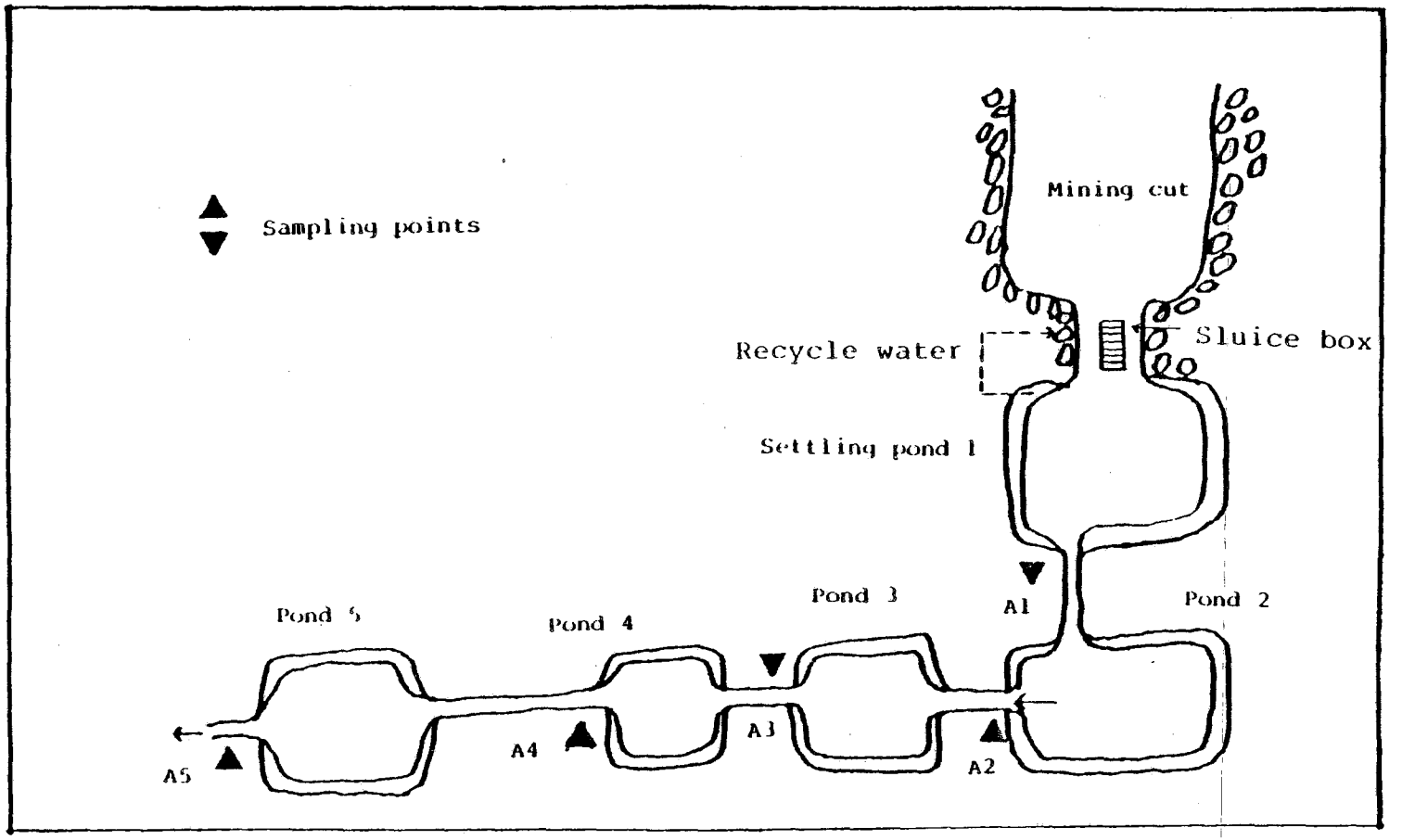


Figure 7. Locations of sampling points.

minutes longer, and record the volume of settleable matter as the milliliter of solids per liter of effluent.

B. Turbidity Measurement

Turbidity in water is caused by a variety of suspended materials with size ranging from colloidal particles to coarse sand or silt. Turbidity analysis in water is based on the optical property of light scattered or absorbed by suspended particles as it passes through a water sample. The intensity of scattering and absorption is dependent upon the size, shape, and refractive index of the particle. Among these, particle size plays the most important role. The smaller the size, the greater the surface area which results in greater light scattering and causes a higher turbidity reading (Chang, 1979).

There are a number of methods which have been used for the determination of turbidity. In this study a Rt-100 turbidimeter, manufactured by Shaban Manufacturing Inc, Fort Meyers, Florida, with formazin as a standard was utilized and measurements obtained as Nephelometric turbidity unit (NTU). It is recommended by the instruction manual that the most accurate range for this instrument is between 0 to 50 NTU. Therefore, the water sample was continuously diluted and thoroughly mixed until the turbidity reading fell less than 50 NTU. The real turbidity of the sample was calculated by multiplying dilution factors and reading for the diluted sample.

C. Zeta Potential Measurement

Colloids have been classified as hydrophobic and hydrophilic on the basis of their electrostatic characteristic. Compounds such as starch, gums, and protein are hydrophilic, while clays and metal oxides are mostly hydrophobic in nature. It has been mentioned in Chapter III that the stability of these hydrophobic colloidal particles is probably due to the phenomenon of the electric double layer which consists of a charged-particle surface and a surrounding sheath of counter ions (Van Olphen, 1977).

Within the diffuse layer, there is a shear plane which represents the portion of bound ions that will remain attached to the colloidal particles as they move through the dispersion. The bound-ions layer can be considered as an internal part of the charged-particle surface. The zeta potential, expressed in terms of millivolts, is defined as the potential required to transport a unit charge from the bulk solution to the shear plane. Its magnitude is considered as a measure of the repulsion between colloidal particles. The higher the zeta potential, the higher the repulsive force will be.

Zeta potential measurement was conducted by a Zeta-meter, produced by Zeta-Meter Inc. of New York, New York. The water sample was subjected to centrifuging to obtain a more diluted supernatant for ultramicroscopic observation. If the stability and destabilization are due primarily to the electrostatic force, this analysis might be helpful in the study (Riddick, 1961).

3. Mineralogical Analysis

Different clay minerals such as kaolinite and illite will present specific properties in dispersion because of imperfection substitution factors and difference of structure. In his study of Florida phosphate slimes, Lamont (1975) indicated that clay minerals should have strong correlation with settling characteristics. Therefore, the clay and colloidal particles were subjected to X-ray diffraction analysis to identify their mineralogical composition.

With the help of a centrifuge, ultrafine particles were collected and then ground into fine powders. The fine material was mounted on a glass slide with water, and scanned over the appropriate 2 theta range using a RIGAKU diffractometer. Upon finishing the analysis, angles 2 theta could be transferred into d-spacing and mineralogical identification was accomplished.

4. Chemical Composition Analysis

A DCP spectrometric analysis is a convenient method to identify the major elemental constituents of solid samples. The procedure of sample solution and preparation is listed in Appendix B. After the completion of sample solution, it could be analyzed by a spectrometric Spectraspan IV plasma emission spectrometer to reveal the elemental composition.

5. Flocculation Test

In water treatment, an optimum flocculation process represents the achievement of equilibrium among involved factors such as pH value, solids content, mixing speed, temperature and chemical dosage. At the present time, we are not able to predict the optimum conditions on the strength of theory. Thus flocculation is a kind of state-of-art process, and only can be determined experimentally.

Determining the optimum dosage conditions can be accomplished by (1) the standard jar test (Hammer, 1970; O'Brien, et al., 1977), (2) Zeta potential measurement (Riddick, 1968), (3) the standard graduated cylinder method, and (4) the flocculation series test (Van Olphen, 1977).

With regard to reproducibility, neither the standard graduated cylinder method nor the flocculation series test can avoid being criticized for their manual processing. Regarding the zeta potential, because PEO is a nonionic reagent, there will not likely be a substantial change in surface charge. A modified jar test recommended by Eckenfelder, Jr. in 1970 was employed to substitute for the

normal jar test process. Instead of using multiple stirring devices for their low stirring speed, (100 RPH), a magnetic stirrer is utilized in this study to obtain a thorough mixing in a short period of time.

In all the water samples, the slurry was placed in beaker on a magnetic stirring device with an 1" spin-bar. The diluted PEO solution was added to the water sample from a burette. The residual turbidity was measured by a turbidimeter to measure the effect of the various factors that affect flocculation performance. The procedure for the flocculation test is described in Appendix C.

CHAPTER V

RESULTS AND DISCUSSION

Using the procedures described in Chapter IV, the experimental results will be listed and discussed in this chapter. The data of particle size distribution will be plotted into "Equivalent Spherical Diameter" vs. "Cumulative Mass Percent" diagram. The factors which will affect the performance of flocculation also will be charted for the purpose of more thorough illustration.

Experimental Results

1. General Characteristics

A. pH Value Measurement

A pH of approximately 7, or near neutral is required for normal aquatic growth and fish survival. Changes in pH may also affect the solution of trace metal in water. From Chang (1979), it was noted that the pH value of both Livengood and Harrison areas was from neutral to slightly basic range (7.6 to 8.6), while in this study, the pH values are from slightly acid to neutral range (6.6 to 7.3). In general, the mining operation does not have substantial adverse effect on pH value in water downstream. The results of pH value at Gilmore and Crooked areas are listed in Table 3.

B. Solids Content and Specific Gravity Measurements

Solids, regardless of being suspended or settleable, will contribute to turbidity. Settleable solids will settle under quiescent conditions because of the influence of gravity. This is the function of a settling pond. From the result of this study, the solids content were found to be continuously decreasing from sample A1, 3.23% to 0.32% in sample A6. For the samples from Crooked Creek area, B1 and B2, there is some substantial difference in solids content. Because the decreasing slope is much less sharp after sample A2, it seems that settling ponds were only effective for particles of certain size, and are thus unable to reduce turbidity to the required standard.

The results of solids content and specific gravity assays are presented in Table 3. It is also noticed that the samples from Crooked Creek area have lower solids content than the samples from Gilmore Creek area and this might have effected flocculation performance.

C. Particle Size Distribution Analysis

Settling ponds have been the most common means of

Table 3. General Characteristics of samples, A1 to A6 from Gilmore Creek area, B1 and B2 from Crooked Creek area.

Residual Solids Sample	Specific pH	Imhoff Cone Content %	Turbidity Gravity	Test ml/L	NTU
A1	6.6	3.23	1.028	0.7	30,000
A2	6.6	1.35	1.008	<0.1	15,600
A3	6.7	0.96	1.006	<0.1	12,000
A4	6.9	0.72	1.005	<0.1	8,900
A5	6.8	0.66	1.004	<0.1	8,100
A6	6.7	0.33	1.001	<0.1	2,700
B1	7.3	1.90	1.007	0.4	3,200
B2	7.4	1.53	1.004	<0.1	2,800

treating placer mining effluent in Alaska (USEPA, 1985). It has been estimated that more than two-thirds of the Alaskan operations use settling ponds (cited in Johnson, 1985). The reason for the popularity is that they can be economically built with the equipment on site, and there is no large initial investment of materials or time as required in some other treatment plants.

As predicted by Stokes' Law and settling column tests, settling ponds are able to achieve 78 and 87% removal of total suspended solid respectively (R and M, 1982). But actually only 60% removal can be obtained in real operation. Therefore, R and M Consultants Inc. reported that the efficiency of removal in a pond is primarily dependent upon the percentage of fine particles (clays and silts) smaller than 20 microns. Johnson (1985) made a similar conclusion that even the well designed ponds with a retention time of days were still unable to remove the particles less than 10 microns in diameter.

It was mentioned in a previous chapter that samples of slurry need to be dried, filtered and weighed to make appropriate samples for size distribution analysis in the Sedigraph analyzer. The amount of particles larger than 60 microns in diameter filtered off is tiny in both samples. It is also remarkable that the size of nearly all particles is less than 10 microns. The results are shown in Figures 8 to 13. Figures 8 to 11 represent the size distribution of samples A1 to A4, and Figures 11 and 12 for samples B1 and B2 respectively.

Figure 10 illustrates that all the sizes of particles are less than 1 micron in diameter for sample A4. As a result, no analysis was conducted for sample A5 and A6 owing to equipment limits. The results show that settling ponds still have an effect on the sedimentation behavior of particles less than 10 microns, if the residence time has not been taken into account. It also can be concluded that samples from Crooked Creek area contain a higher percentage of fine particles than the samples from Gilmore Creek area. For instance, sample B2, from the end of the first setting pond, contains 93% of particles less than 1 micron, while sample A1, from the outflow of the first pond at Gilmore Creek area, contained only 73%.

2. Physiochemical Properties

A. Imhoff Cone Test

It is not enough to picture the sedimentation situation of fine particles only from a solids content and size distribution analysis. From the Imhoff cone test data shown in Table 3, the difficulty of fine particles settling is easily understood. Even for sample A1 and B1, the settleable volume is still less than 1 ml/L for one hour settling time. This result explains why most settling ponds are unable to reduce turbidity to the required standard point.

B. Turbidity Measurement

Turbidity is an expression of the optical properties that cause light to be scattered or absorbed rather than transmitted in straight lines through a water sample. It is thus a measure of light transmission and not a measure of total suspended solids. It is, however, used as a surrogate measurement for sediment concentration because it is a convenient tool.

Turbidity can be measured as Jackson turbidity units (JTU) by using a Jackson candle turbidimeter. Since the lowest turbidity limit is 25 units for the Jackson candle method, a Nephelometer was introduced to measure lower turbidity values. The Nephelometer measures the intensity of light scattered by particles in the direction at right angles to the incident light, and the measurements are expressed as Nephelometer turbidity units (NTU). The NTU and JTU are not comparable owing to the difference in equipment operation principles, but can be correlated by comparative data.

The results of turbidity measurements are also shown in Table 3. It simply illustrates that more solids content will cause higher turbidity for the sample from same area.

C. Zeta Potential

Although the zeta potential has been subject to criticism for a period of time for its ill-defined character, it is still a reference in evaluating stability of fine particles in dispersion. Riddick (1968) indicated that negative zeta potential values less than -14 millivolts (mv) always represent the onset of agglomeration, while those which range from -14 to -30 mv mean a plateau region of slight flocculation.

The zeta potential measurement for samples from Gilmore Creek and Crooked Creek area are -16 and -21 mv respectively. Within this range, particles will not readily contact each other automatically due to repulsive forces.

3. Mineralogical Analysis

In his semiquantitative X-ray analysis, Maneval (1985) discovered that there was a great deal of amorphous matter in the samples which were from Interior Alaska placer mines. Some common clay minerals such as illite and kaolinite were also identified in several samples. But in this study, x-ray diffraction analysis indicated the only clay mineral present is kaolinite for both samples. Since the diffraction intensity is quite low and the peak is obscure, it can be deduced that amorphous material is predominant in both samples. The diffraction pattern is shown in Figure 14.

4. Chemical Composition Analysis

Because of isomorphous substitution factors and layer structures, clay minerals are capable of accommodating several cations from their exterior environment. These

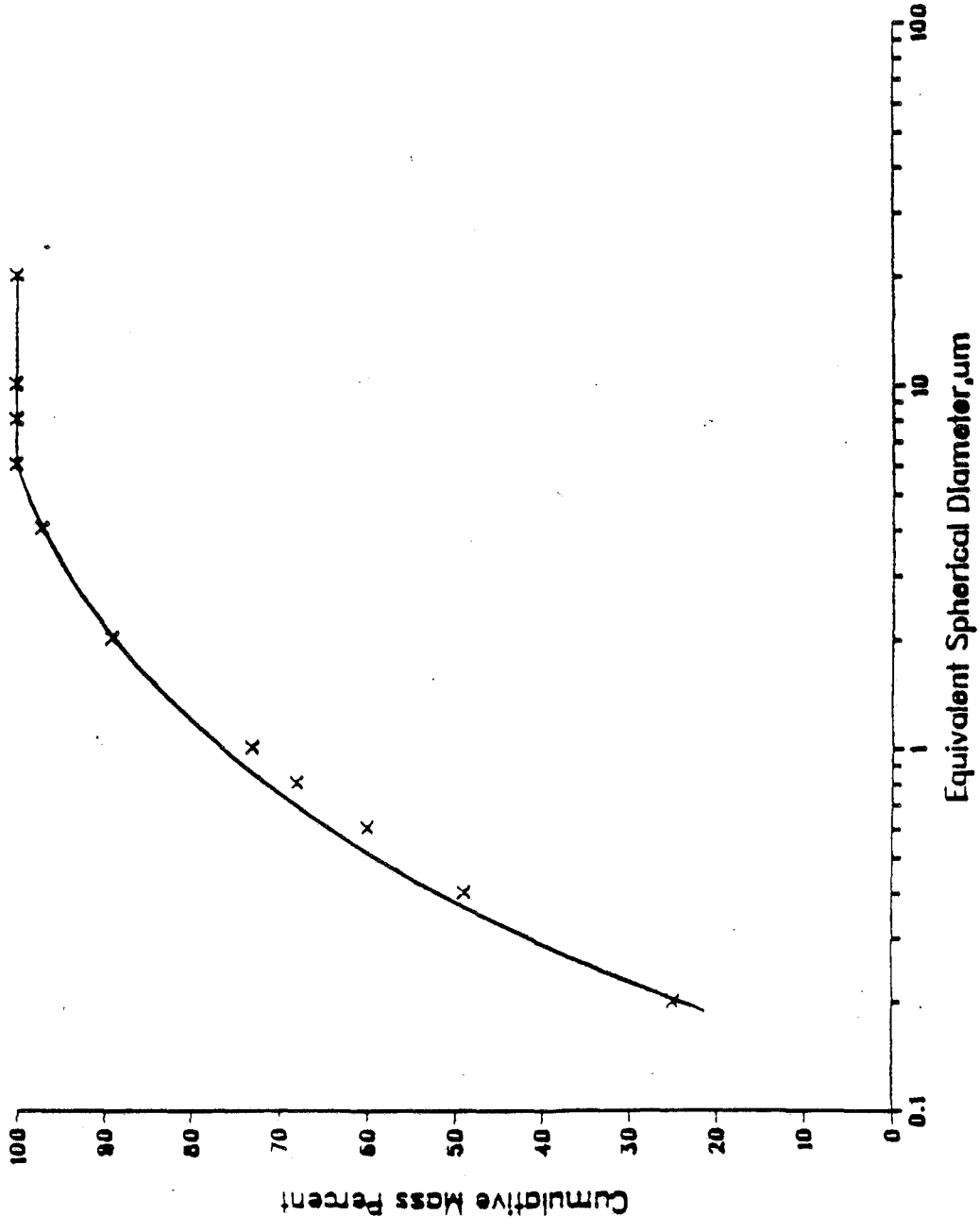


Figure 8. Size distribution of sample A1.

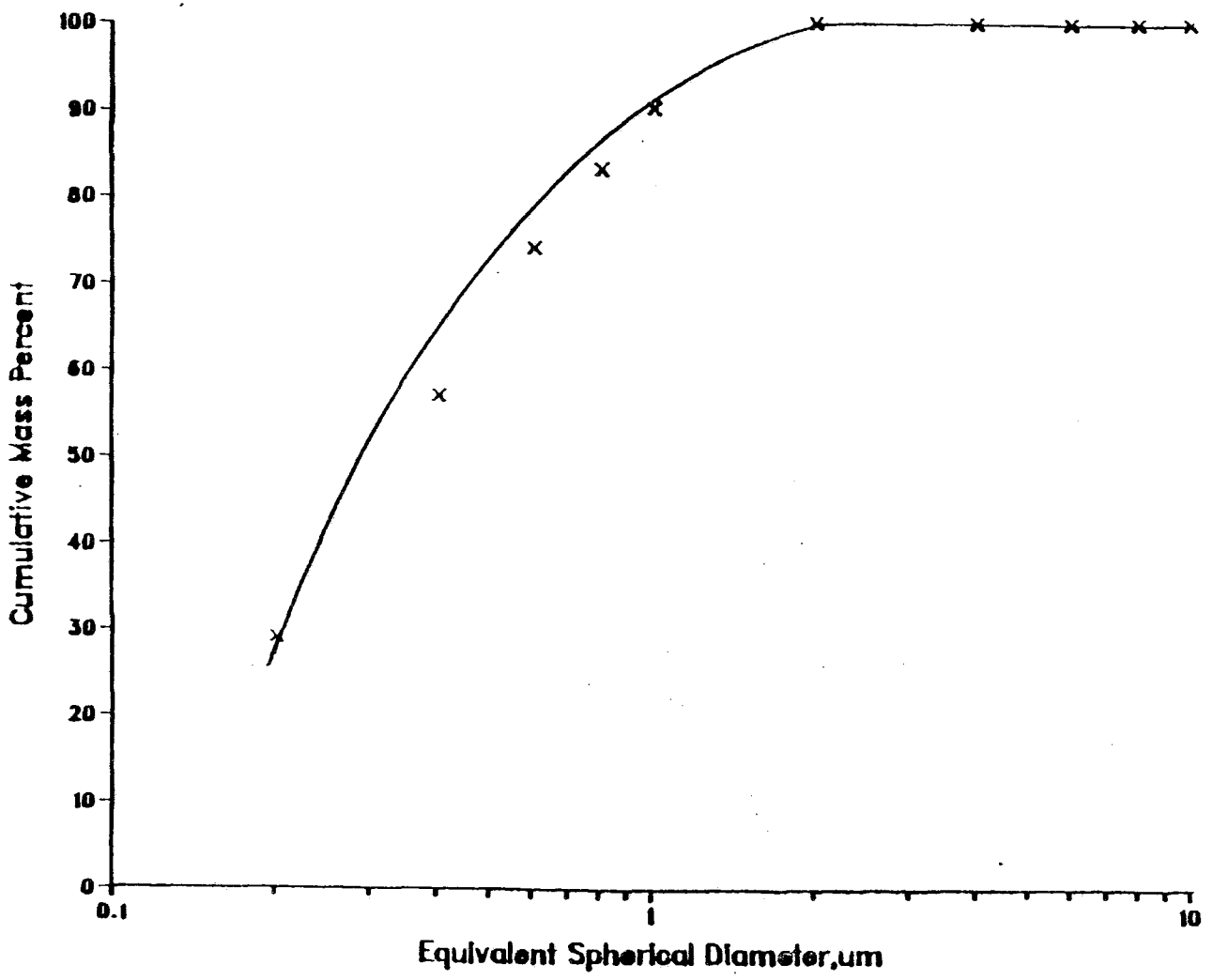


Figure 9. Size distribution of sample A2.

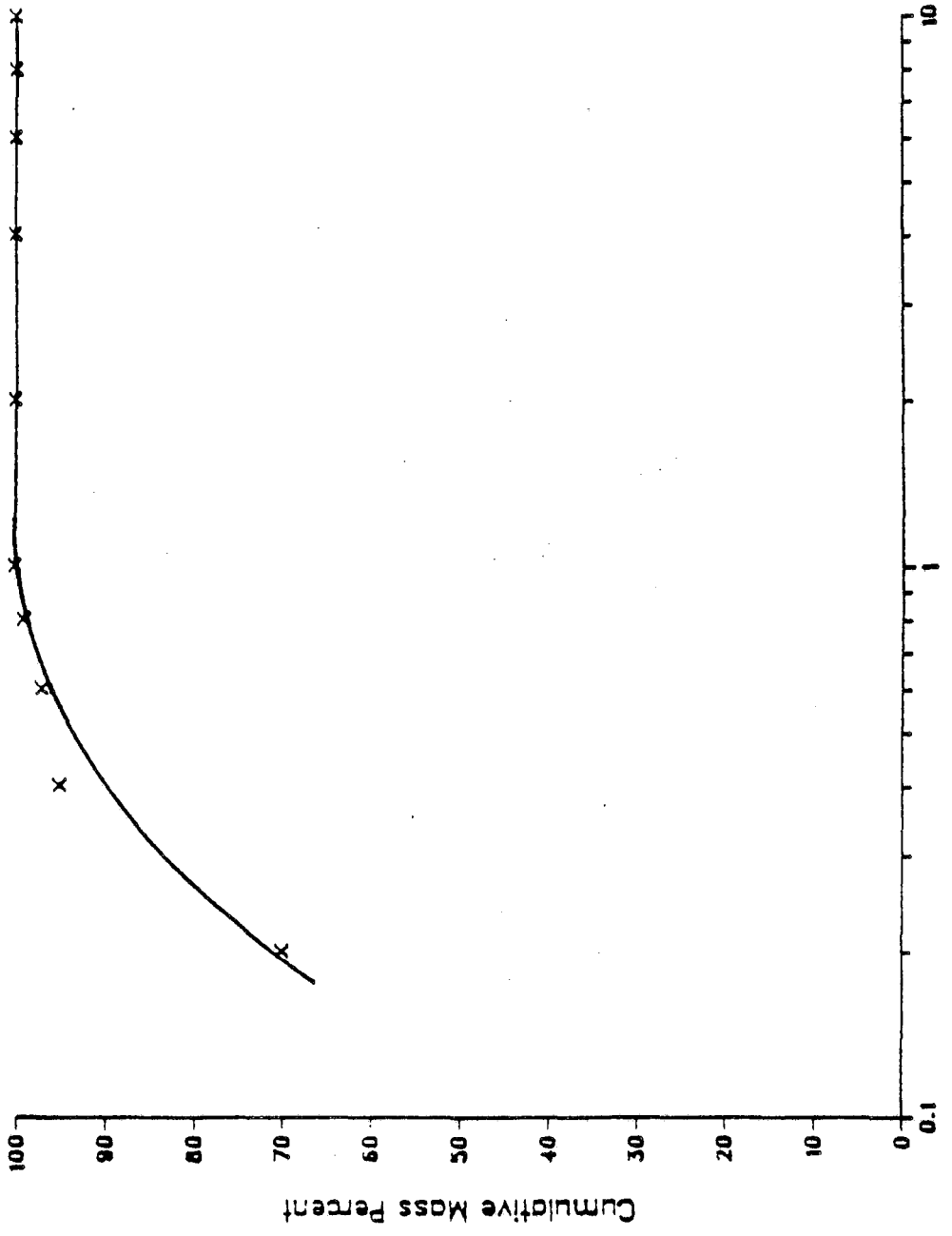


Figure 10. Size distribution of sample A3.

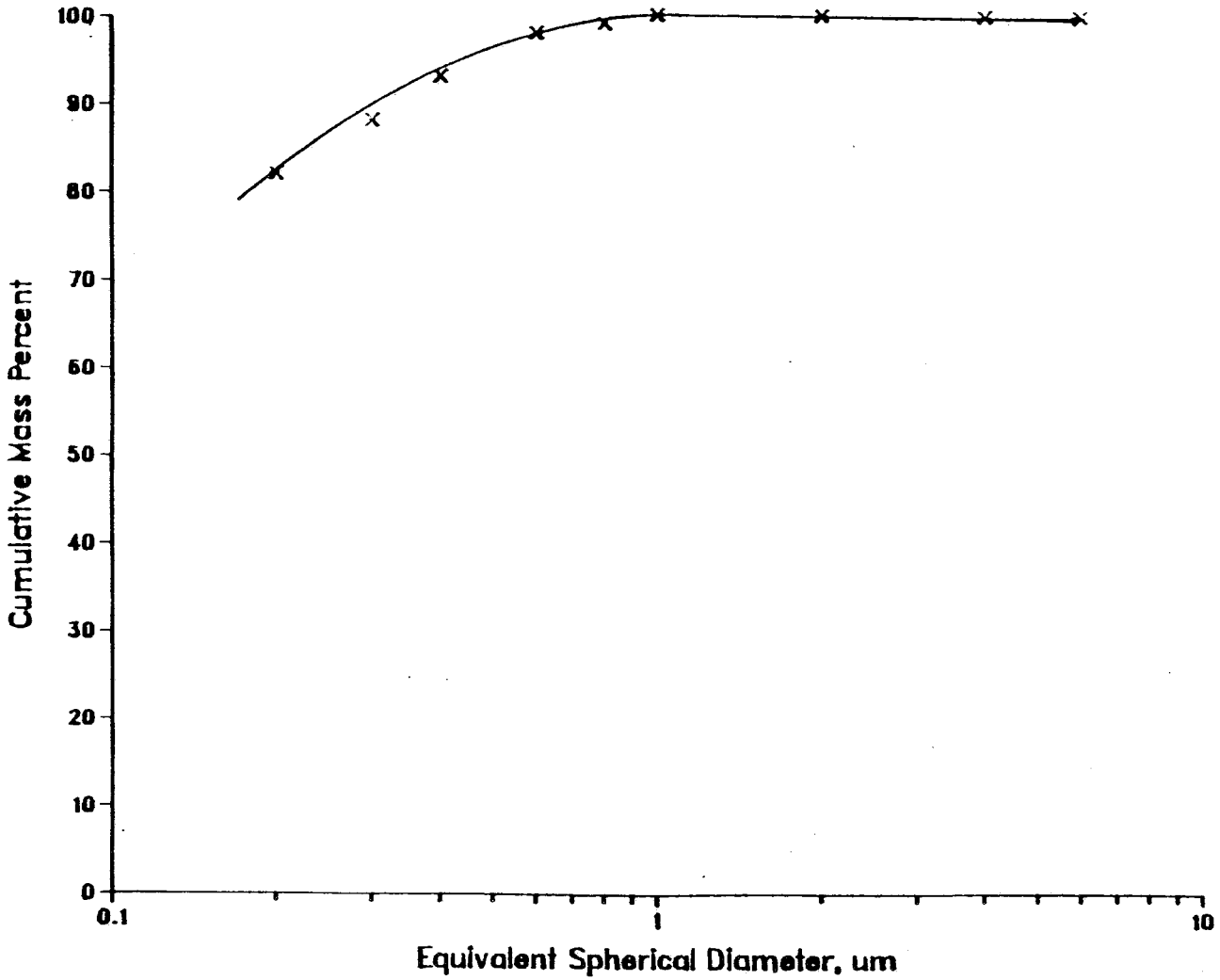


Figure 11. Size distribution of sample A4.

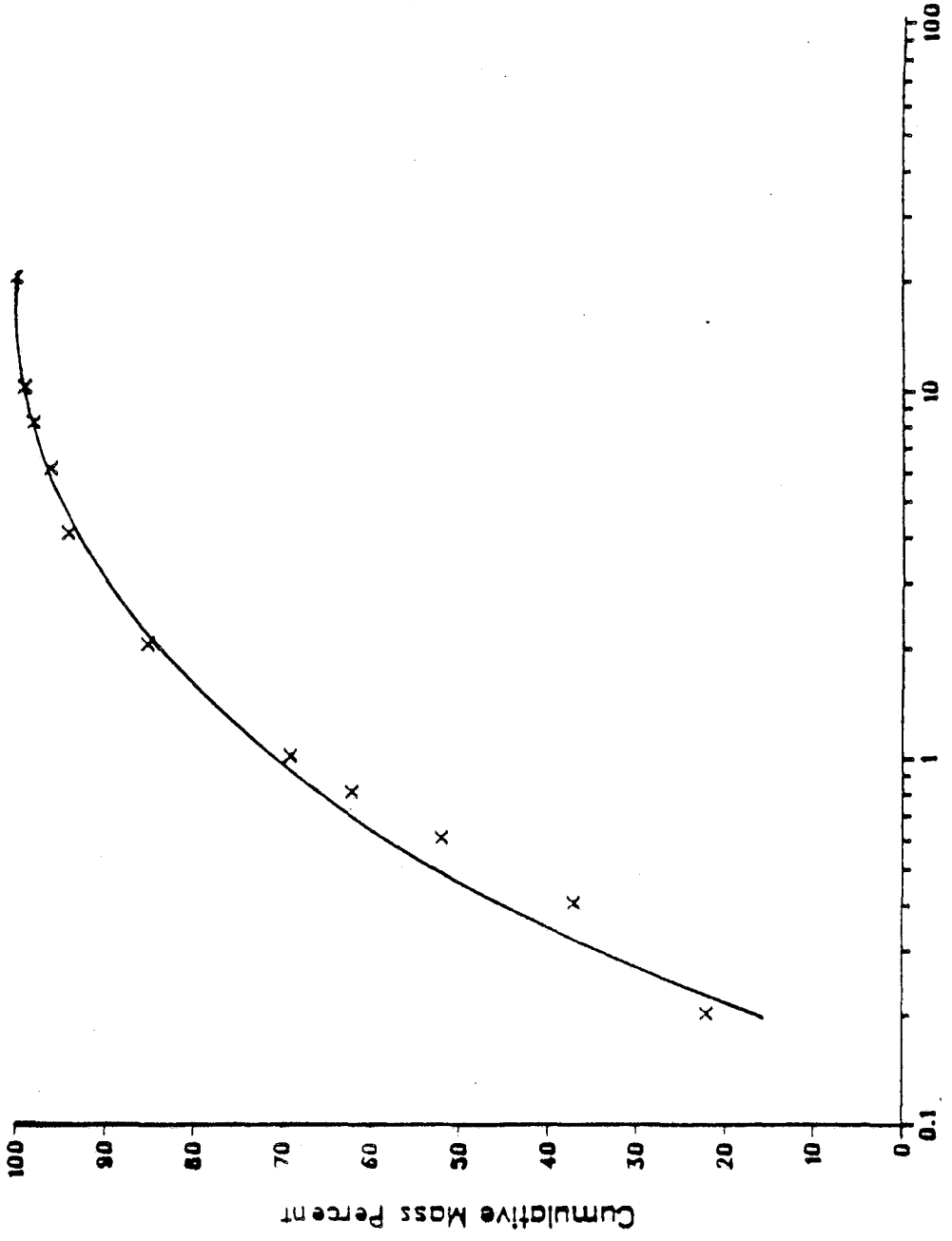


Figure 12. Size distribution of sample B1.

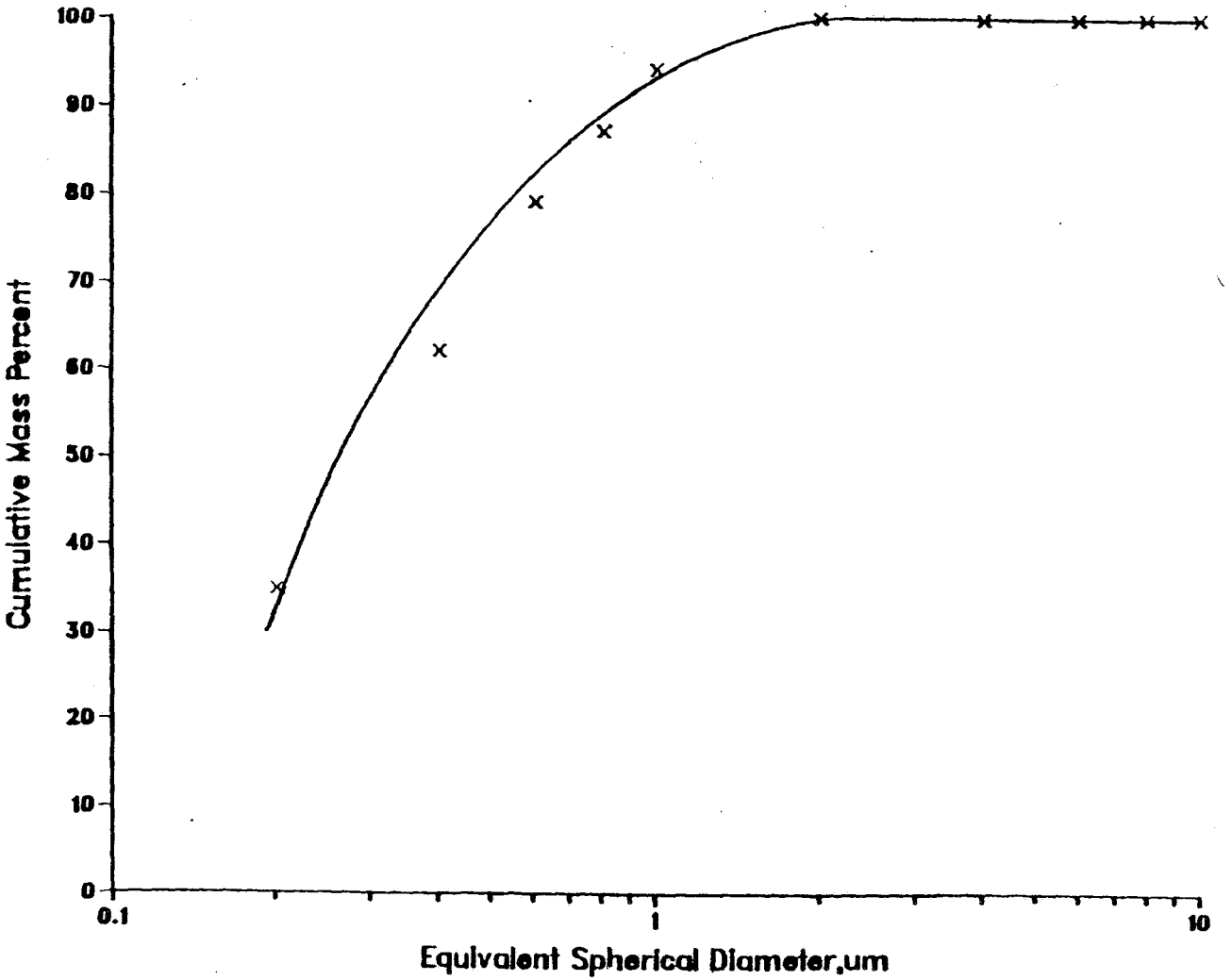


Figure 13. Size distribution of sample B2.

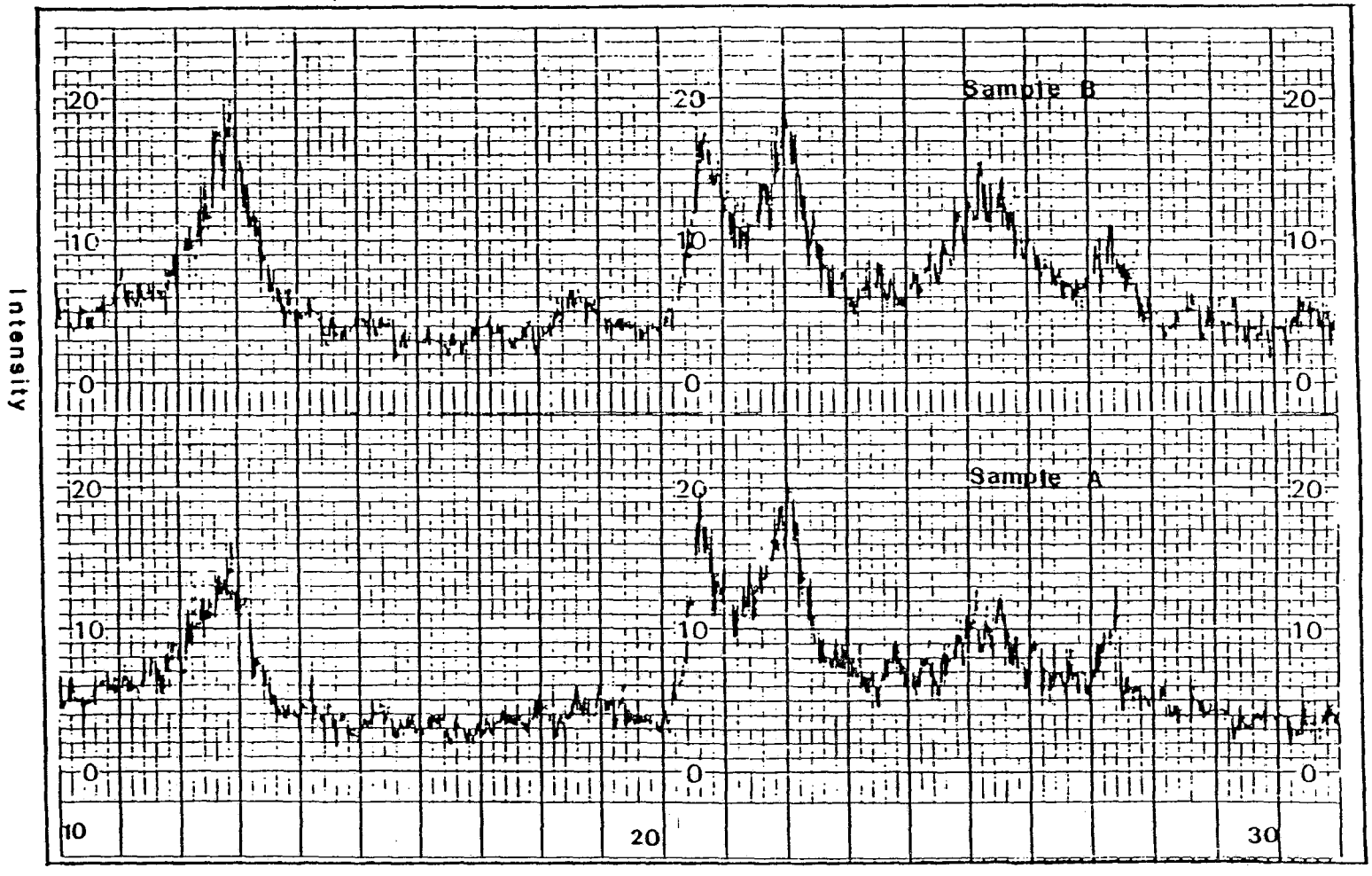


Figure 14. Mineralogical composition analysis by X-ray diffractometer.

cations have a significant effect on the behavior of clay minerals particularly in liquid dispersion. Therefore the DCP is a useful tool for revealing composition which influences dewatering phenomenon of clay particles.

Table 4 shows the chemical composition of samples from Gilmore and Crooked Creek areas. The Table indicates that the sample from Gilmore Creek has more divalent and less monovalent absorbed cations than the sample from Crooked Creek. This seems to be a major cause of different behavior between the two samples.

5. Flocculation Tests

Since a variety of factors are involved in the flocculation process, it is nearly impossible to create optimum conditions theoretically. Thus the determination of the best performance must be conducted step-by-step with experiments. In this section, some variable parameters, which are likely to be important in the flocculation process, were evaluated in laboratory tests.

A. Mixing Speed and Time

Through study of previous research of the pertinent literature, the initial concentration and dosages were chosen as 28 ml/L of 0.05% PEO and 28 ml/L of 0.25% $CaCl_2$ to select the best mixing speed and time before the optimum conditions are determined. $CaCl_2$ functions as a flocculation aid in this process. It is clear from Figure 15 that the higher-speed mixing caused lower residual turbidity in the same period of mixing time. And, in general, both over-and under-mixing can greatly reduce the polymer effectiveness. As a result, 9 minute mixing time with a mixing speed 9 on magnetic stirrer scale (400-500 RPM) was determined as standard for the experiments that followed.

Table 4. Results of chemical composition analysis from Direct Current Plasma (DCP).

Gilmore Creek Sample	Crooked Creek Sample Weight %	Sample Weight %
SiO_2	30.6	31.3
$Al2O_3$	20.9	26.7
Fe	18.2	19.7
CaO	4.5	2.0
MgO	3.3	1.4
Na_2O	0.9	1.3
K_2O	1.3	1.8
H_2O	20.3	15.8

B. Settling and Conditioning Time

It is recognized that there is a distinction between mixing and conditioning. Mixing is used to homogenize the dispersion which involves water and applied chemicals, while conditioning (a gentle stirring) is referred to as a treatment by which flocculation of this homogeneous mixture is promoted.

From Figure 16, fast mixing is shown to be more important than gentle agitation or conditioning because conditioning which followed mixing does not apparently enhance the efficiency of flocculation in this case. It also has to be noted that the flocculated flocs settle so fast that there is no remarkable decrease in residual turbidity after a short settling time.

C. Optimum Dosage

Since the efficiency of clay flocculation is known to be inversely proportional to the ratio of the ionic radius to charge (R/C), and the R/C for Na^+ and Ca^{+2} ions is 0.95 and 0.495 respectively, Ca^{+2} ions should be able to increase the performance of flocculation test in this study.

Also from the standpoint of the double layer theory, surplus Ca^{+2} ions will increase ionic concentrations in the solution and thus compress the diffuse layer. As a result, the constant attractive forces, van der Waals' forces, will predominate over repulsive forces and overcome the energy barrier to enhance the formation of aggregates. Therefore, $CaCl_2$ can be used as an aid in clay flocculation technology.

The results of this study is in accordance with the conclusion mentioned above. Without $CaCl_2$, the residual turbidity can only be reduced to 90 NTU or so regardless of the amount of added PEO solution. But with the adding of $CaCl_2$ prior to PEO, the turbidity is able to be lowered to 20 NTU. Sample A5 clearly projects a picture of the function of $CaCl_2$ in Figure 17. It also has to be noted in Figure 17 that polymers which over-saturate the available surfaces of the dispersed particles will produce a restabilized colloid, since no sites are available for the formation of bridges. On the other hand, under-dosage certainly has no capability in achieving the required standards.

The volume of flocs has to be kept in mind as well while choosing the best dosage. If the volume of settled flocs is too large to be controlled by hand, it is not practical in field processing even if some turbidity is permitted. Thus, the concentration and optimum dosage for PEO and $CaCl_2$ are

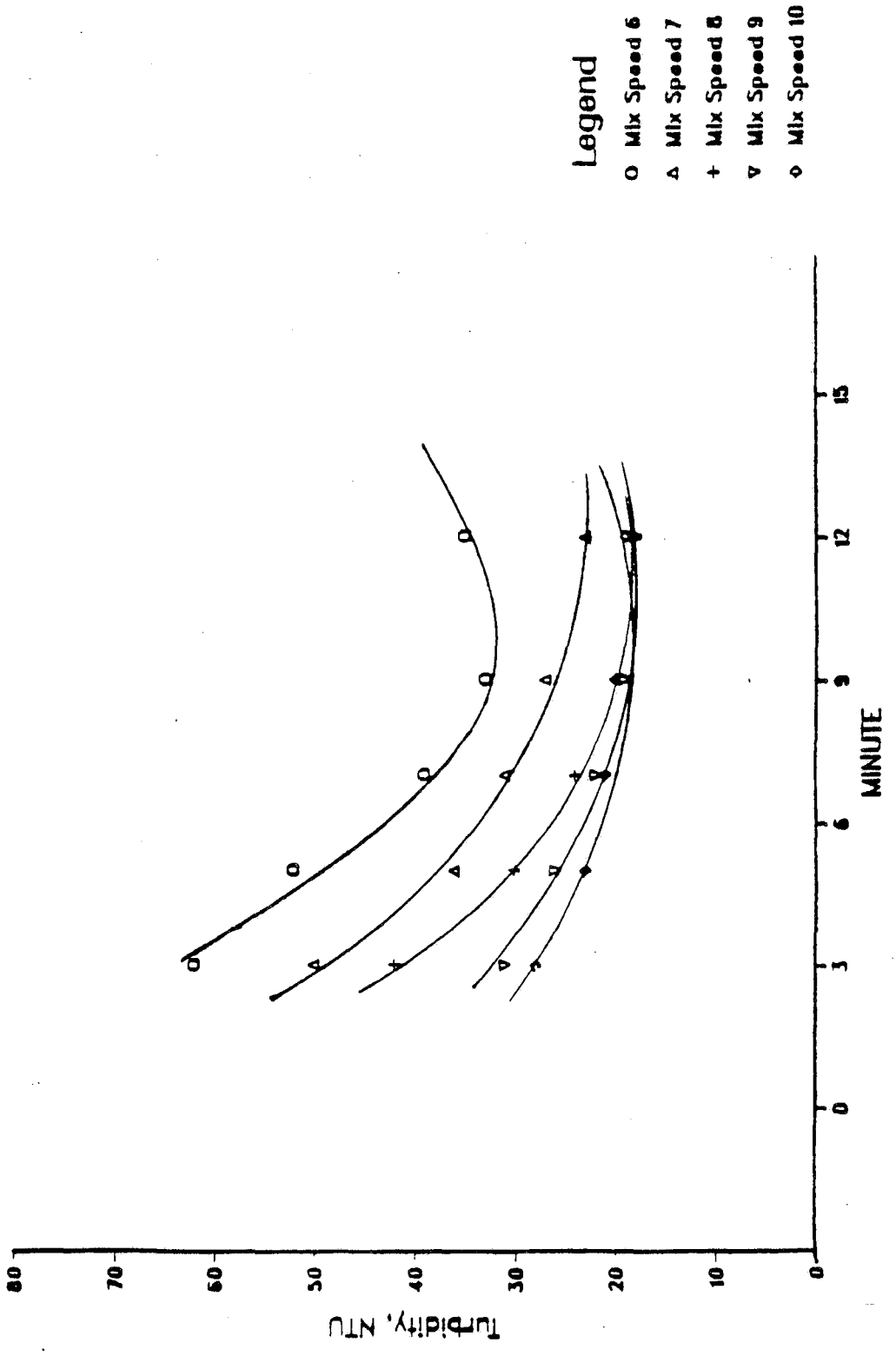


Figure 15. Test of mixing time and speed, 7 ml of 0.05% PEO, 7 ml of 0.25% $CaCl_2$ on sample A (250 ml).

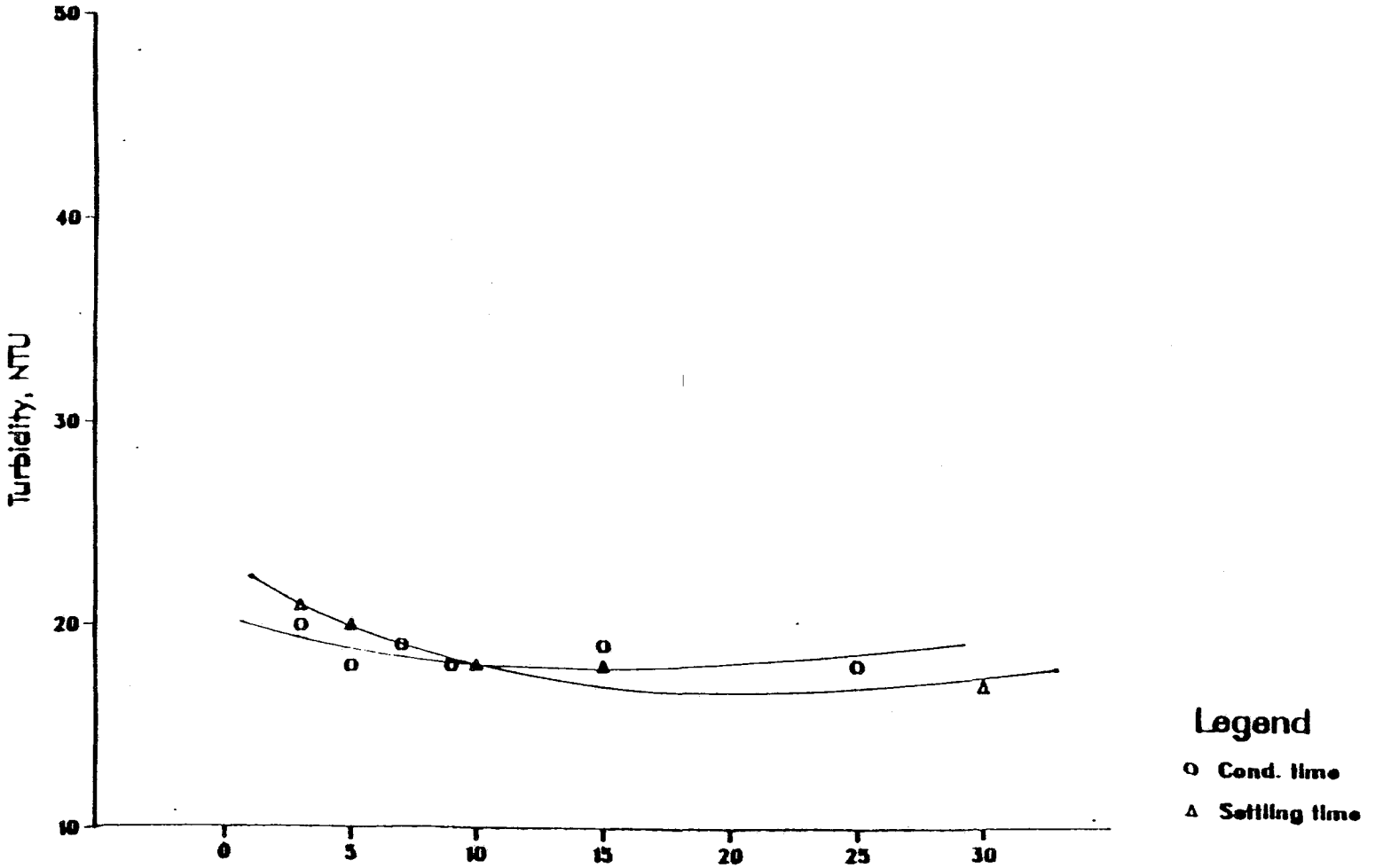


Figure 16. Test of conditioning and settling time, 7 ml of 0.05% PEO, 7 ml of 0.25% $CaCl_2$ on sample A5 (250 ml).

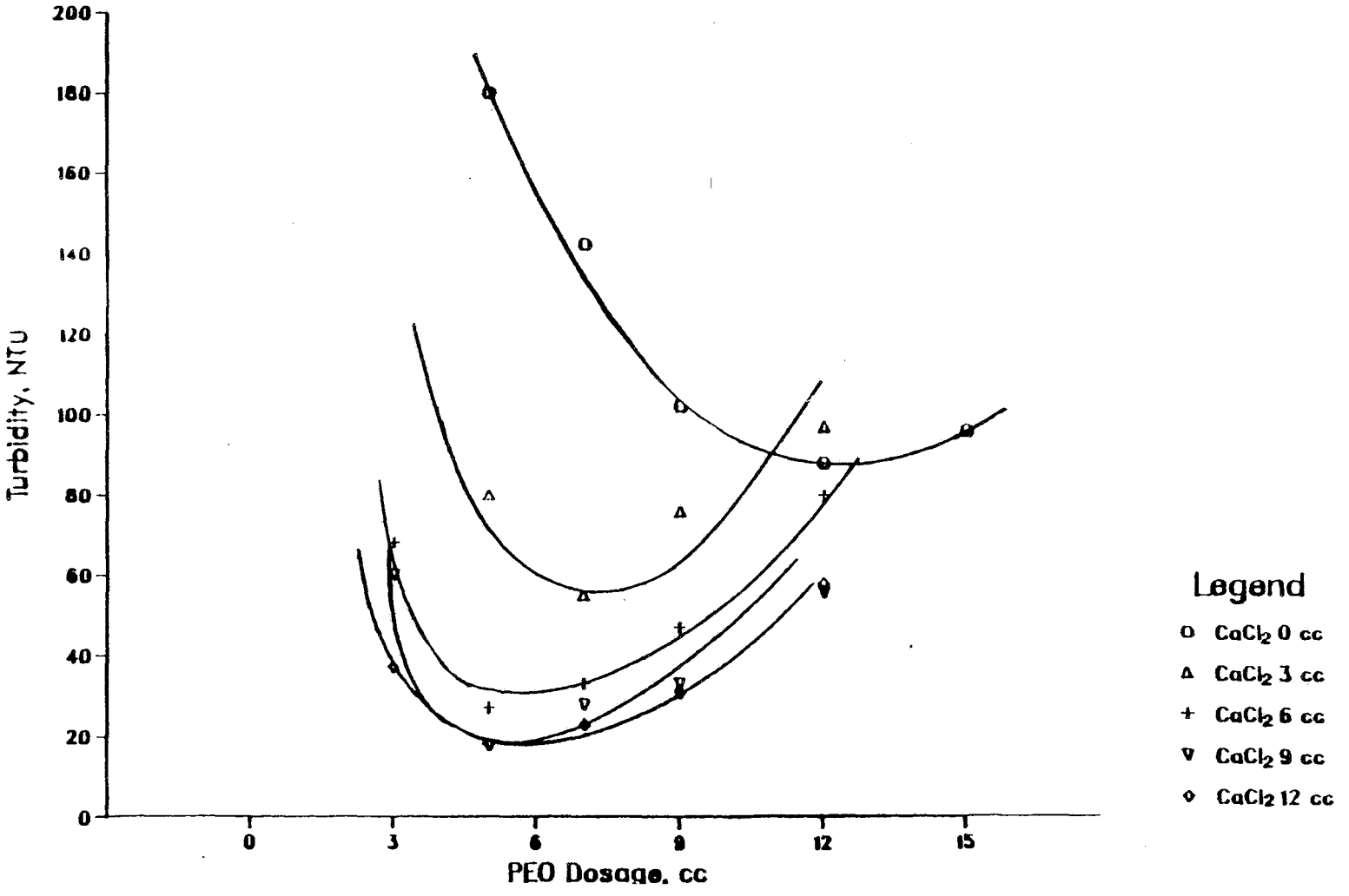


Figure 17. Result of optimum PEO dosage test on sample A5 (250 ml).

chosen as 0.05% 20 ml/L and 0.25% 36ml/L respectively for samples from Gilmore Creek area.

Figures 18 to 20 show the experimental results of sample B2 from Crooked Creek area. In Figure 18, both PEO and $CaCl_2$ are unable to function well enough to reduce turbidity. The lowest turbidity which can be achieved is 70 NTU. In Figure 19, nevertheless, the acid ions play a positive role in performance. It is well known that the edges of clay minerals will carry a positive charge with decreasing of pH (Van Olphen, 1977). In order to enhance the intensity of the positive charge and thereby promote the attraction of edge-to-face, HCl dilution has been utilized in sample B2 for decreasing its pH value. Under the pH value (6.0) from Figure 18, the required turbidity standard may be achieved and the optimum dosage can be chosen from Figure 20.

In the comparison between sample A5 and B2, the size distribution and chemical composition analyses offer important evidence. Sample B2 contains a higher percentage of ultrafine particles than sample A5, thus the edges of particles might perform a more significant role in flocculation processing. The DCP data reveals that sample B2 consists of more Na_2O and K_2O, while sample A5 contains more MgO and CaO. Both Mg^{+2} and Ca^{+2} ions have lower R/C ratio than K^+ and Na^+ ions. Therefore, there is no doubt that sample B2 is more difficult to clarify.

D. Solids Content Factor

Zatko (1982) indicated that PEO can be used for dewatering in a wide range of slurry concentrations and that higher solids content samples consumed larger PEO dosages. Yang (1979), however, concluded that high solids content inflow of a settling pond needed less flocculant dosage than that of low solids content outflow. This controversy will be studied here.

It is known that agglomeration of particles involves two stages: (1) particle movement to make interparticle contact, and (2) particle destabilization to permit attachment when contact occurs. Figure 21 presents the result of three different solids content samples. It indicates that the higher the solid weight-percent, the larger PEO dose that has to be used to achieve the same clarification condition. Also remarkable is that over-dosage more easily occurs for samples which contains lower amounts of solids. It seems reasonable that particle collisions are far less important in the mechanism of polymer flocculation, while they are essential for traditional metal coagulants. The reason is that the particles are connected only by collision in Al(III) and Fe(III) coagulation process because no bridging action occurs.

E. Synergistic Factor

Owing to being a high cost reagent at this time, PEO dewatering technique may be economically unattractive. However, if wide spread use were made of PEO, "economy of scale" probably will lower the cost of this chemical. Another method for improving the economics of utilizing PEO would be to find a low-cost reagent that could be used to replace a portion of PEO.

Smelley and Scheimer (1980) concluded that the synergistic effect is nil when cationic and nonionic polymers were used in combination with PEO. For anionic reagents, even when a synergistic effect was observed when PEO was used with polyacrylamide, there was no advantage over PEO in terms of economics.

Natural guar gum, a nonionic polymer with molecular weight of approximately 220,000, has been recommended by Smelley because it seemed effective in synergism. Figure 22 indicates that the PEO dose can be substantially reduced when gum and PEO are used together. This is also true for sample B2 shown in Figure 23. In addition to the synergistic effect, guar gums seem capable of improving the performing efficiency. With 8 ml/L of 0.1% gum, turbidity could be reduced to 13 NTU by only 8 ml/L of PEO for sample A5. For sample B2 in Figure 23, it has the same dual functions as well. Therefore, guar gums are chosen as synergistic aid for joint use with PEO.

F. PEO Concentration Factor

As a powdered polymer, a stock solution of PEO must be prepared in distilled or clean water before processing can be started. For obtaining a homogeneous solution, a thorough stirring with a magnetic stirrer is necessary. It took about 2 hours to prepare a completely dissolved PEO solution in this case.

For the purpose of shortening the length of preparation time and determining the efficiency of different concentration of solutions, the PEO concentration factor is discussed here. 12ml/L of 0.1% PEO is able to reduce turbidity to the same level as 24 ml/L of 0.05% PEO in Figure 24 for sample A5, but 48 ml/L of 0.025% PEO fails to reach the same turbidity level. This situation also can be noticed in Figure 25 for sample B2. A 40ml/L addition of 0.025% PEO was unable to reduce the turbidity as well as 20ml/L of 0.05% PEO did. As a result, a conclusion can be drawn that although more diluted PEO stock solution is more easily prepared, it might have poorer performance in actual mineral processing. An appropriate concentration should be determined by experimentation.

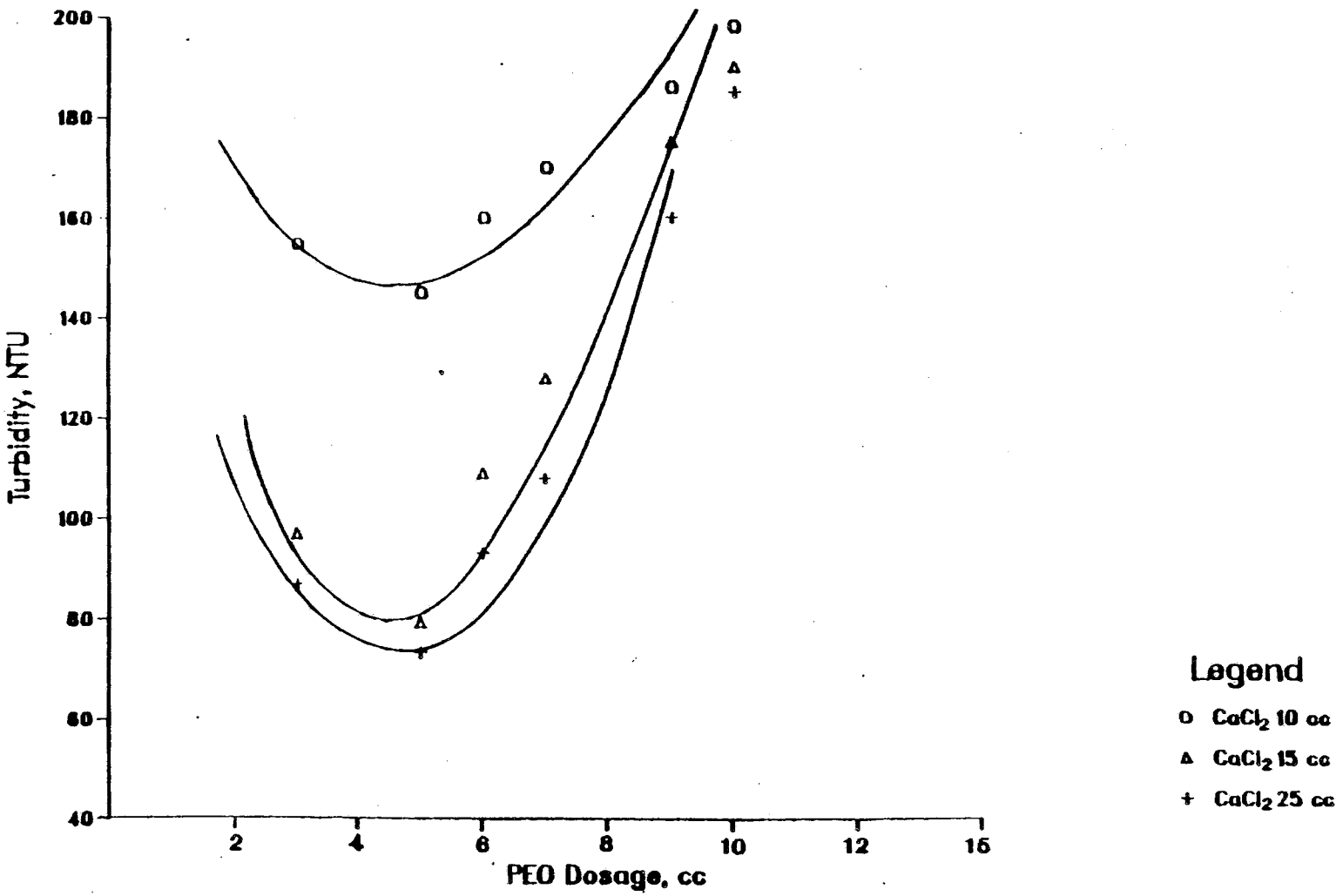


Figure 18. Result of optimum PEO dosage test without changing pH on sample B2 (250 ml).

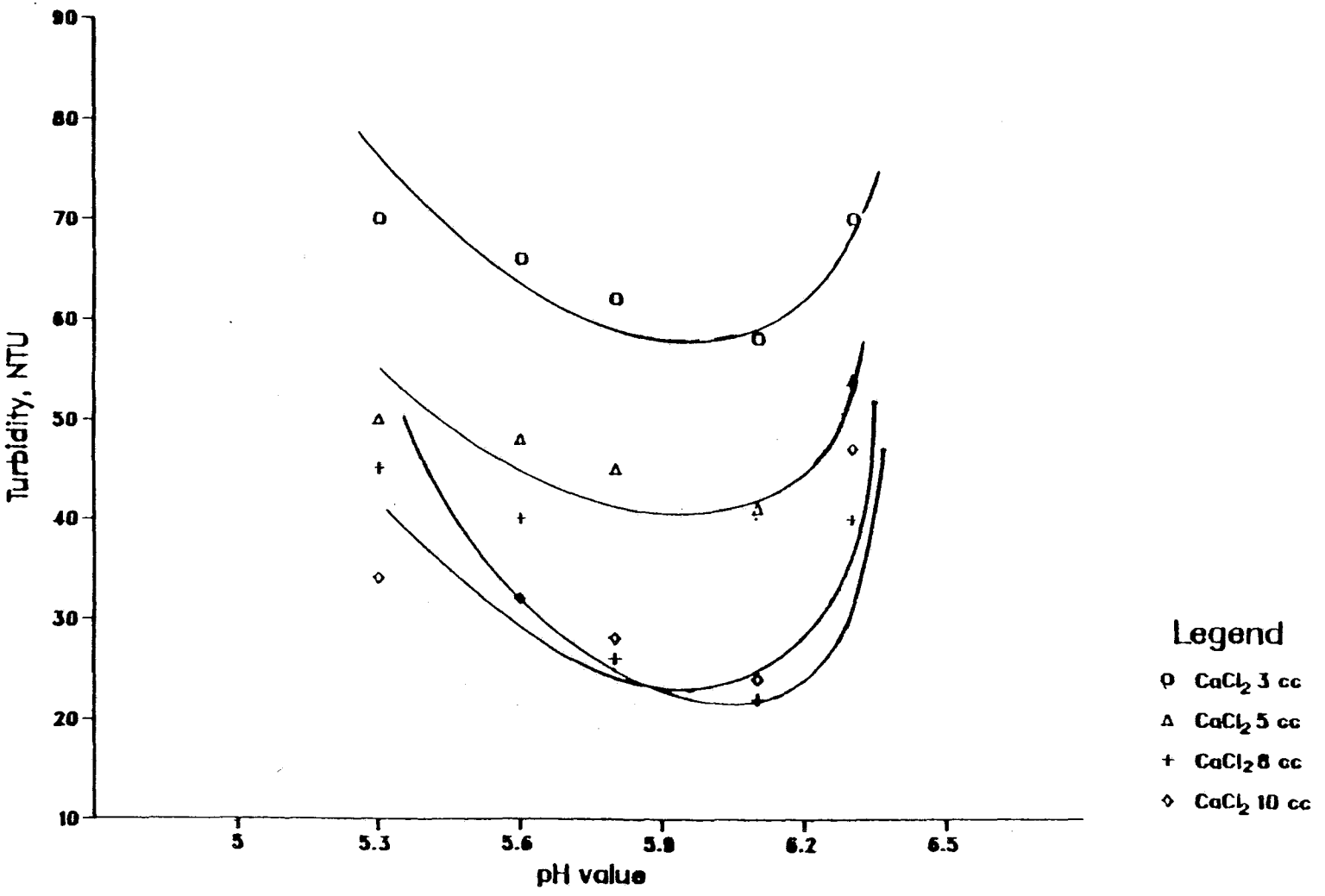


Figure 19. Result of different pH under 5 ml of 0.05% PEO on sample B2 (250 ml).

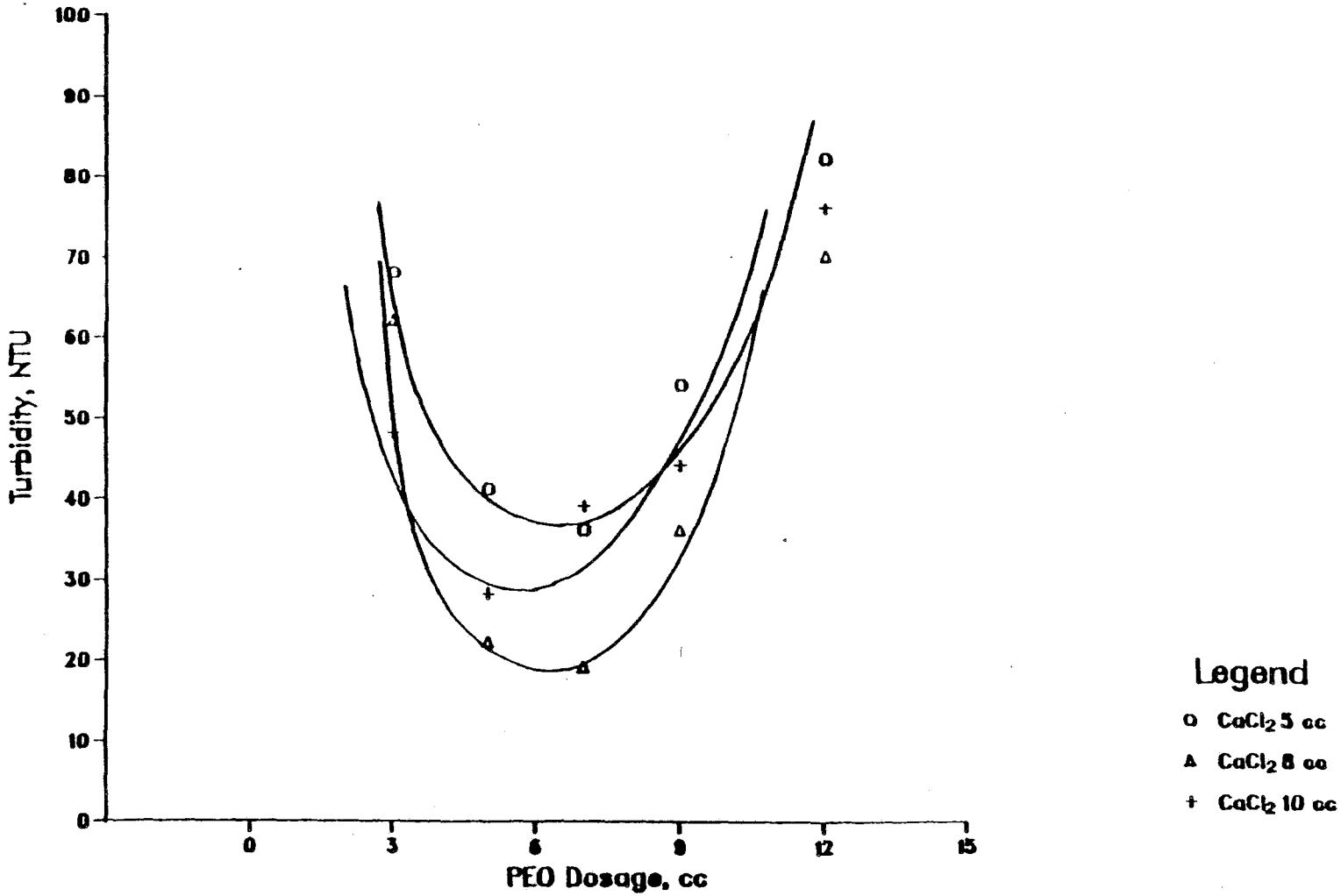


Figure 20. Result of optimum PEO dosage test under pH 6.0 on sample B2 (250 ml).

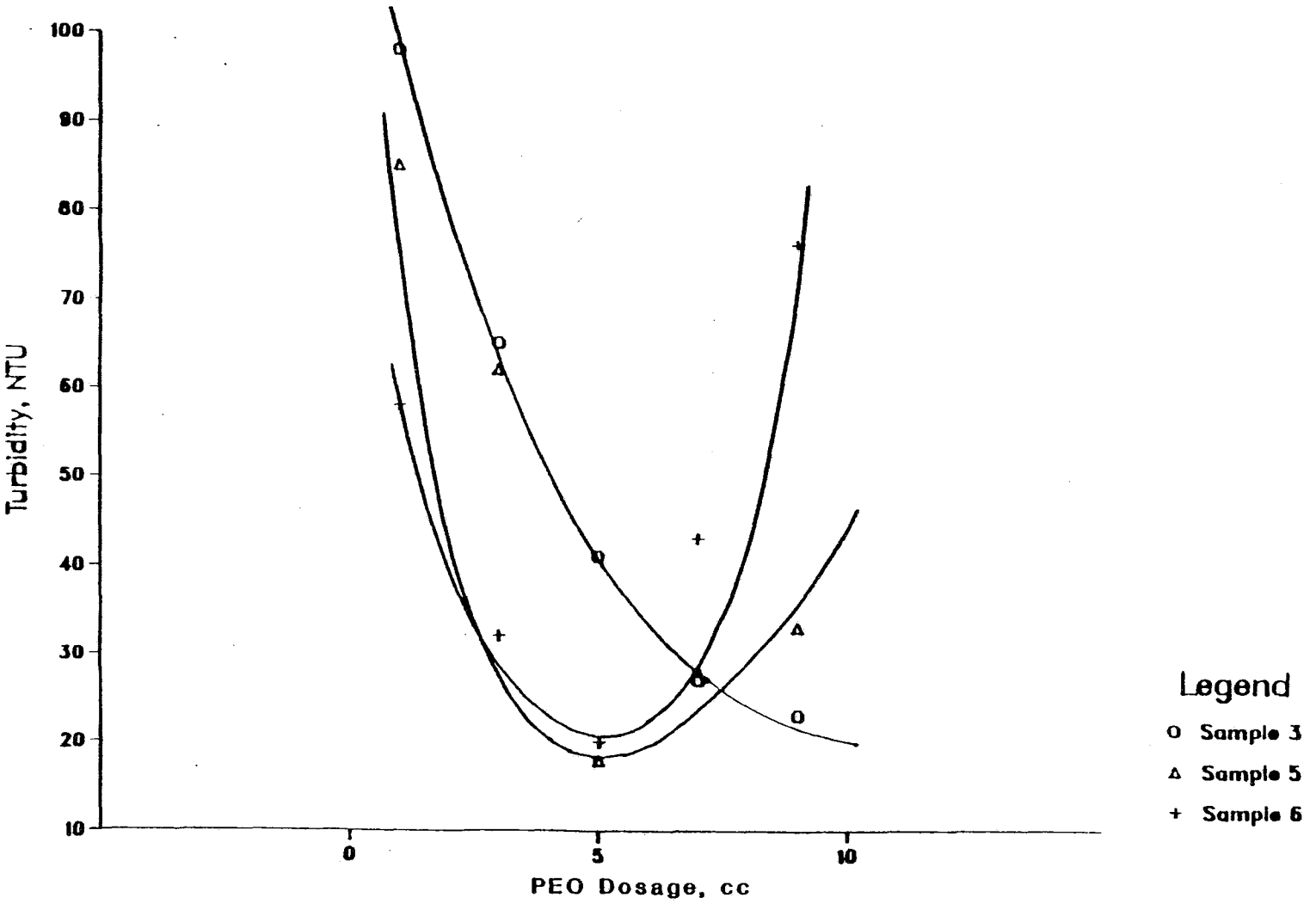


Figure 21. Solids content effect on flocculation process, sample A3, A5, A6 (250 ml).

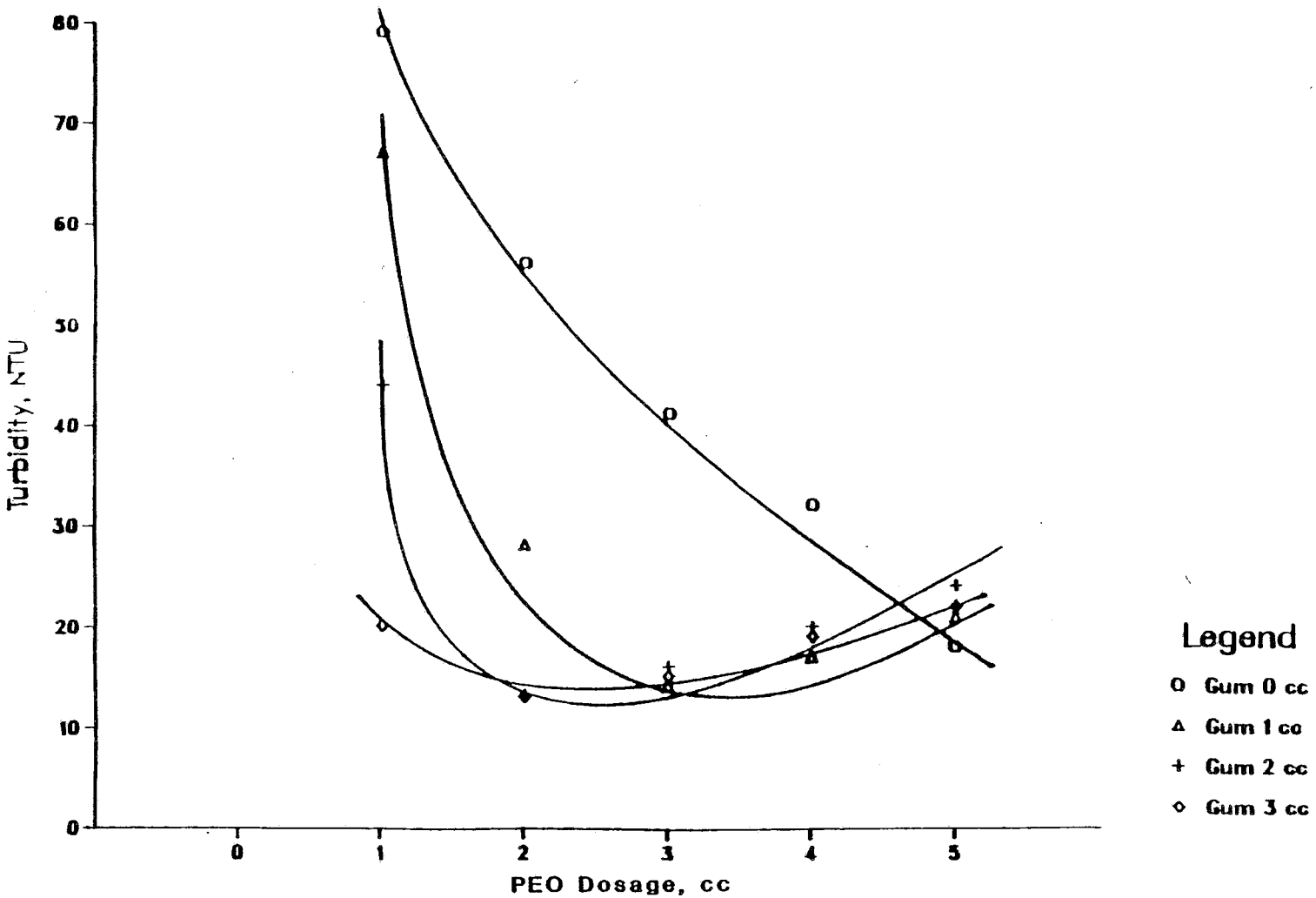


Figure 22. Synergistic effect of guar gum on sample A5 (250 ml).

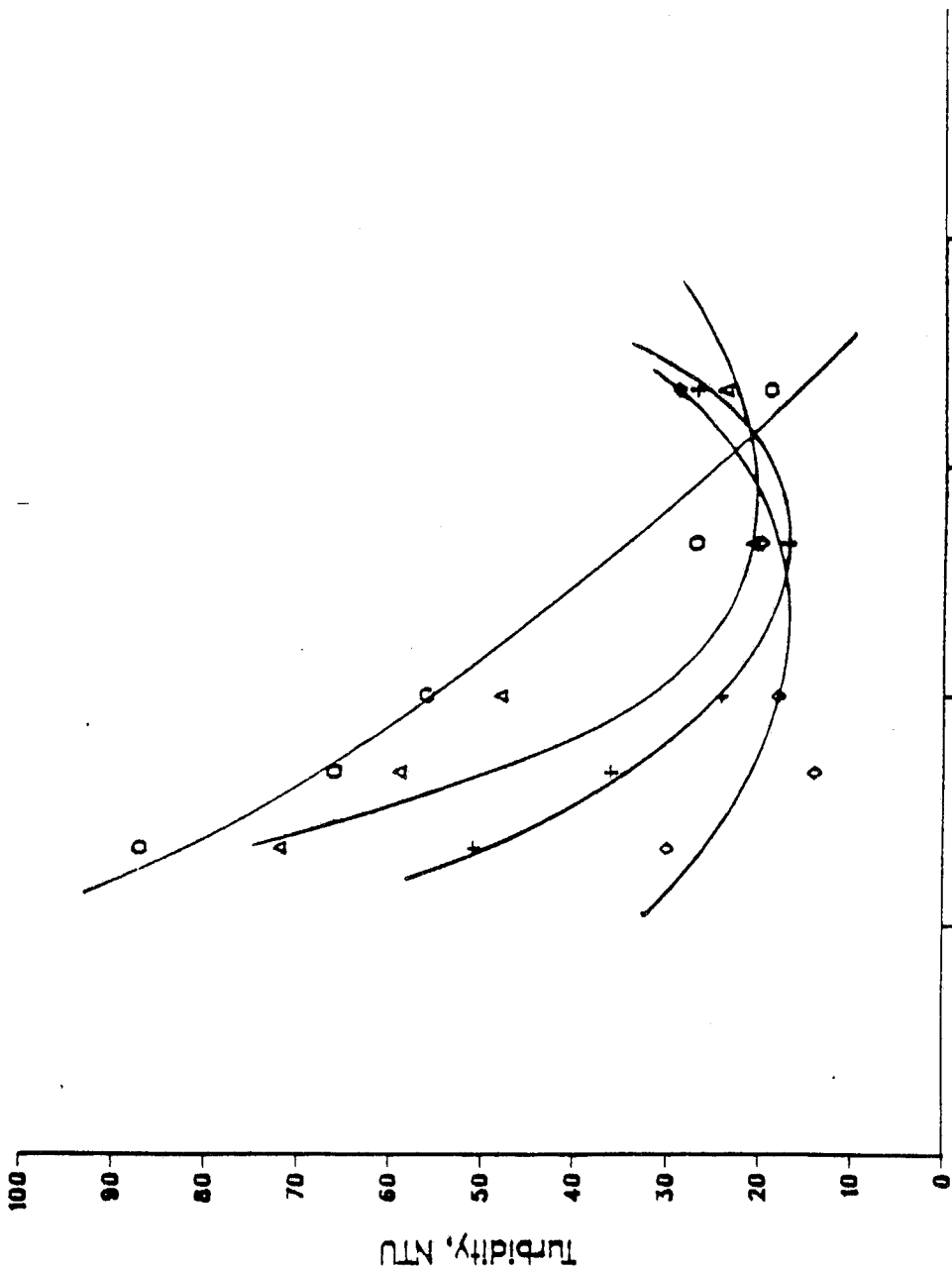


Figure 23. Synergistic effect of guar gum on sample B2 (250 ml).

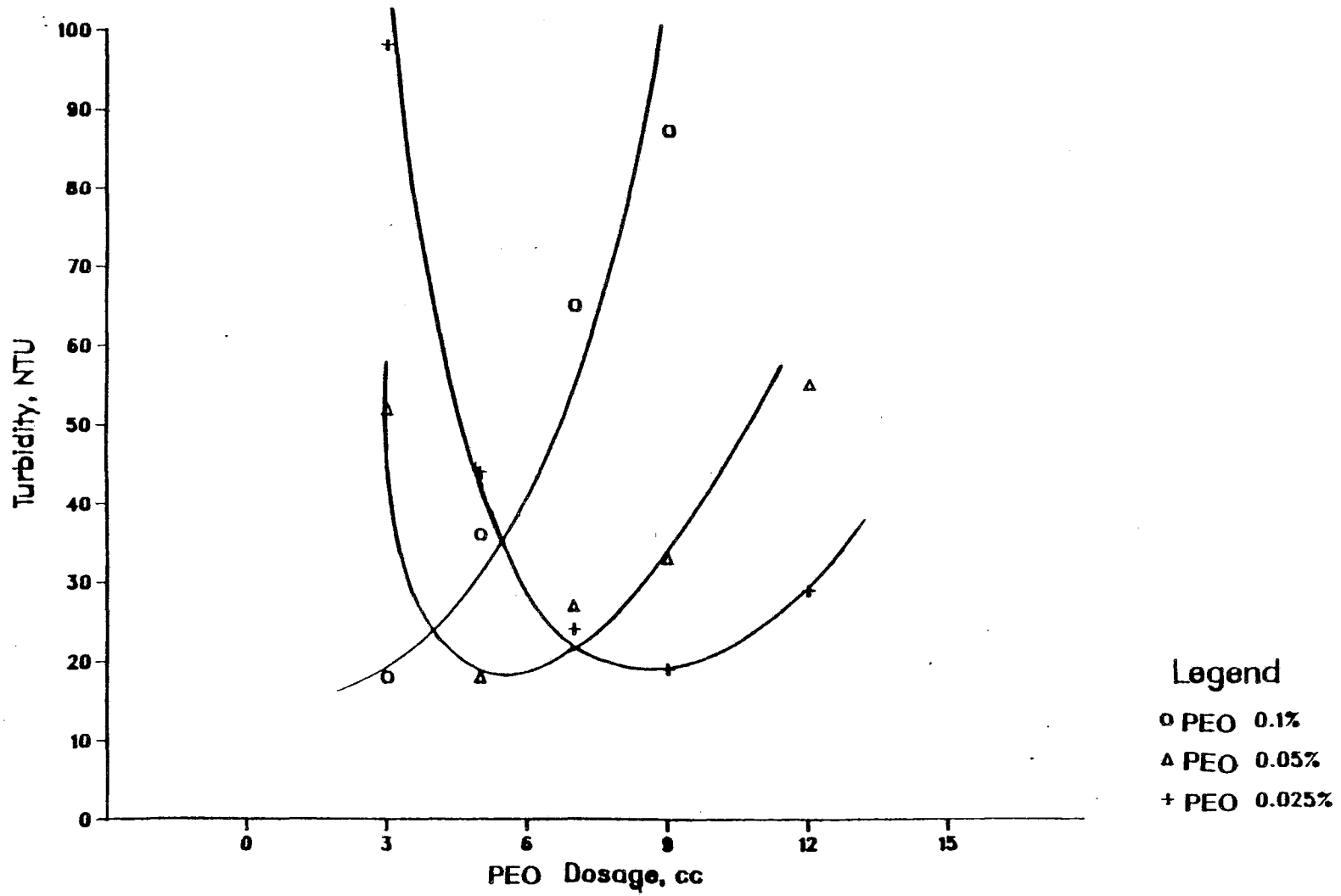


Figure 24. Effect of PEO concentration on flocculation process of sample A5 (250 ml).

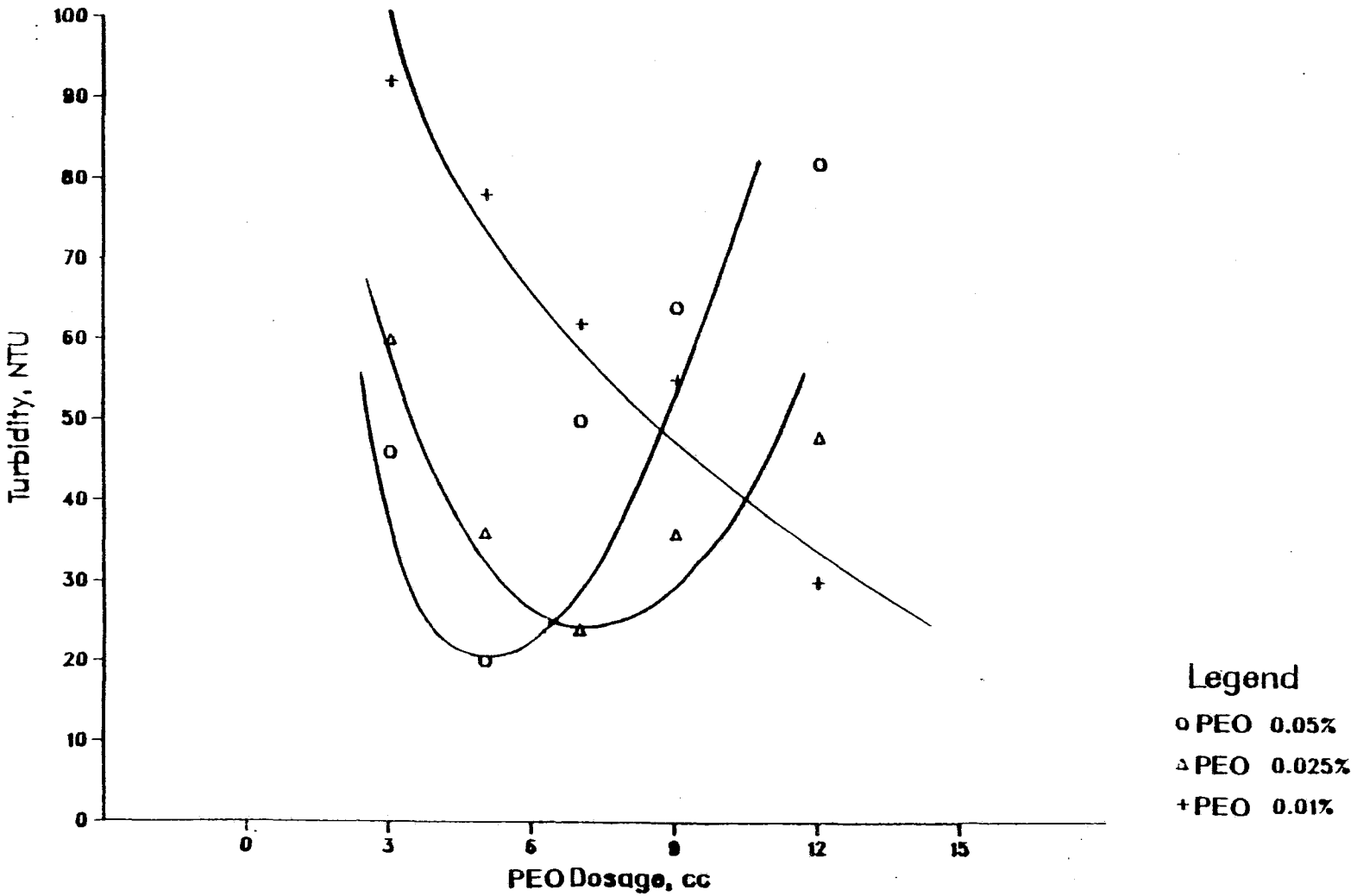


Figure 25. Effect of PEO concentration on flocculation process of sample B2 (250 ml).

CHAPTER VI

PLANT DESIGN AND ECONOMIC EVALUATION

Following laboratory experiments, a field plant should be designed and evaluated to study the feasibility of the PEO flocculation process. Before a well designed plant can be achieved, economically and practically, there are two features which must be considered. Since the settling pond is the most popular and inexpensive technique, it has to be included in order to get rid of settleable particles, thus reducing the consumption of PEO. Another is the technique of recycling which will lower the amount of wastewater that has to be treated by a chemical process. Therefore, the only effluent that needs treatment is the surplus wastewater to be discharged into a receiving stream.

Recycling

Of major concern to placer miners has been the belief that there would be loss of fine gold when recycled water contains a high percentage of solids. This is motivated by concern that the change of specific gravity and viscosity will affect the settling behavior of fine gold. Lin (1985), however, indicated that no gold loss occurred using sluicing waters containing solid concentrations up to 107,000 mg/L. Shannon and Wilson (1985) have studied the effects of various total suspended solids (TSS) and viscosity levels on fine gold recovery. They concluded that no gold loss happened at TSS levels ranging from 0 to 195,000 mg/L, and that exceptionally high levels of TSS (>20 weight percent) might have effects on migration of fine gold. These high levels have not yet been measured in actual field conditions.

Settling Ponds

Even if the normal solids content of process water has been proved not to have an adverse effect on gold recovery, it should still be reduced to a point as low as possible to eliminate any risk of fine gold loss in the sluice box, and also lessen the PEO dosage. In order to gain the greatest efficiency in settling ponds, several factors should be taken into account such as: (1) retaining water long enough for coarse particles to settle, (2) preventing of short-circuiting, (3) minimizing scour and resuspension of solids during periods of high flow, and (4) preventing washout during flood events (Weber, 1985).

Chemical Treatment Plant

After a well-designed settling pond has been constructed, the recycled water will have very low solids content that can minimize the negative effects on gold recovery. Then a chemical treatment plant recommended by Smelley and Feld (1979) can be designed and fabricated based on the amount of surplus wastewater. It consists of a slime holding tank, a reagent holding tank or tanks, a conditioner-mixer with a propeller-agitator to mix slime and PEO and/or other solutions, and a rotary screen to air dewatering and consolidation of the solids. Also included are two or more positive displacement pumps to feed the slurry and the flocculant at a predetermined rate.

The slurry is pumped into the conditioner-mixer to mix with the prepared PEO solution from the reagent tank. Mixing of both PEO solution and slurry can be performed by a straight-blade propeller mounted on a central shaft driven by a variable-speed motor at the top of the shaft. There are outlets for the overflow to be transported to the dewatering trommel. A fine opening screen for promoting adhesion of the flocculated material lines the first part of the trommel, the whole trommel also would have to be fitted with a larger opening screen for accomplishing the later dewatering operation. The discharged water can be drained to another polishing pond through a ditch. The function of this pond is to settle slow forming flocs which leak out from the screen of the trommel, this thus would guarantee the water to be discharged into the receiving stream will meet the required standards. The cleaned water can be used to mix with recycled water to reduce solid content, and it can be applied to dilute the stock PEO solution as well. The dewatered solids have the function of enhancing the walls of settling ponds. A view of this process is shown in Figure 26.

Since the equipment size is primarily dependent upon the amount of surplus wastewater that has to be handled, miners ought to conduct an individual calculation to design an effective plant. Smelley and Feld (1979) reported that for a 30 gal/min output, the equipment list is as follows:

Slurry holding tank	— 1000-gallon capacity.
Reagent holding tank	— 20-gallon capacity.
Conditioner-mixer	— 100-gallon capacity.
Rotary screen trommel	— 2' diameter x 15' (48-mesh opening at first part).

For promoting efficiency, several factors such as speed and slope of trommel, as well as feeding rate of slurry waste have to be considered in detail. Also important is the retention time of the slurry in the conditioner-mixer and in the trommel.

Economic Evaluation

The optimum concentration and dosage for PEO, $CaCl_2$ and guar gum is 0.05% 8 ml/L, 0.25% 36 ml/L and 0.1% 8 ml/L respectively for samples from the Gilmore Creek area. This reagent consumption can be converted to 0.03 lb

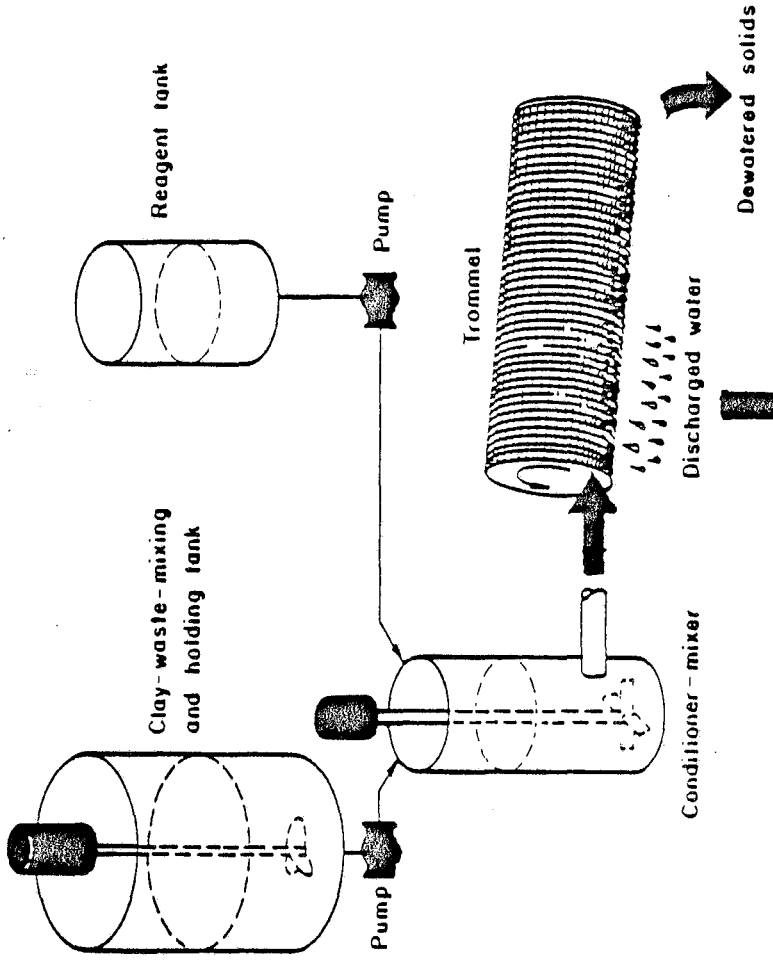


Figure 26. Design of PEO wastewater treatment plant.

PEO, 0.67 lb $CaCl_2$ and 0.06 lb guar gum respectively, required for treatment of 1000 gallons of wastewater. Chemical costs can be calculated under the optimum conditions and following assumptions:

Average water consumption: 3,000 gal/min

Recycled water percentage: 80% (2400 gal/min)

Treated water percentage: 20% (600 gal/min)

Operating hours per day: 10 hours

Operating days per year: 150 days

The chemical costs are shown in Table 5, and the total chemical cost per year based on the preceding assumptions is $17,000. For equipment cost, Smelley et al. reported that a mixing and dispersal system for PEO would be $15,000 to $20,000 for an average operation. Here neglected are costs of labor, utilities, etc., which should be taken into account to perfect a treatment design.

Table 5. Chemicals Cost Evaluation (Using Optimum Amounts of Chemicals)

	Assuming cost per pound	Consumption per 1,000 gal. (pounds)	Cost (dollars) per 1,000 gal.
PEO	4.47	0.03	0.14
$CaCl_2$	0.20	0.67	0.13
Guar Gum	0.70	0.06	0.04
Sub total			$0.31

CHAPTER VII

CONCLUSIONS AND RECOMMENDATIONS

Conclusions

In conjunction with settling ponds, use of PEO to treat effluent from placer mining has been proved as an effective method, economically and scientifically, in this study.

The solids content found in the wastewater studied here is very low ranging from 3.23% to 0.05%. Most of the particles are less than 10 microns in diameter, and carry -16 to -21 mv of zeta potential. Although placer mining operations normally do not have an adverse effect on pH value, those ultrafine particles are hard to settle and thus cause severe turbidity problem.

The effectiveness of PEO treatment of placer mining slurry can be affected by mixing speed and time, content of fine particles as well as dosage and concentration of reagent solution. Also significant are physical properties and chemical composition of fine solid particles. The flocs settle very rapidly so that no conditioning and settling period is needed for further clarification. The most important fact is that $CaCl_2$ is useful to promote the bridging process, but is not adequate for the whole operation of solid removal.

Optimum reagent dosages for the most difficult to treat waters at the Gilmore Creek and Crooked Creek sites are given in Table 6.

A synergistic function was discovered while using PEO with natural guar gum. It not only reduces the dose of PEO up to 50%, but also improves flocculation performance.

In selecting the optimum dosage of reagents, a modified jar test can be conducted. From mixing speed's point of view, this method has been evidenced to be more effective than the general jar test instrument. A plant can be designed and costs can be determined after finishing laboratory experiments.

Recommendations

1. Temperature effect, a controversial factor which may have an effect on the flocculation process, should be studied in order to evaluate its impact on performance.

2. Besides $CaCl_2$, other flocculation aids such as lime, should be tested to identify the most efficient and inexpensive clarifiers, and the PEO clarifier should be further tested to determine effectiveness under field conditions.

3. The treatment should be run at several placer mining sites for determining difference in individual cases.

4. The mineralogical composition should be analyzed more precisely or quantitatively to uncover the possible correlation between clay minerals and the flocculation process.

5. The mechanism of flocculation can be further thoroughly studied by means of an electron microscope or other equipment.

6. Finally, a detailed site specific economic evaluation should be performed for providing more information at candidate mines.

Table 6. Reagent dosages found to give optimum results in reducing turbidity in Gilmore Creek (A5) and Crooked Creek (B2) mine water samples.

	Chemical Dosage per 1000 gallons					
Sample	PEO Only	PEO + $CaCl_2$		PEO + $CaCl_2$ +Guar Gum		
Gilmore Creek (A5)	0.19 lb	0.08 lb	0.70 lb	0.03 lb	0.67 lb	0.06 lb
Crooked Creek (B2)	NA	0.09 lb	0.62 lb	0.05 lb	0.30 lb	0.07 lb

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APPENDIX

APPENDIX A
Summary Excerpts from EPA Proposed Effluent Limitations

PROPOSAL

Subcategory	BPT	BCT	BAT	NSPS=BAT
<20 yds³/day	No limitations	None	None	
>20 yds³/day to 500 yds³/day all mines	0.2 ml/L SS 2,000 mg/L TSS (Simple settling)	BCT=BPT	BAT=BCT=BPT	
all exc. large dredges > 4,000 yds/day	same as above	Process water- no discharge (recycling) excess water- 0.2 ml/L SS 2,000 mg/L TSS (simple settling)	BAT=BCT	
dredges > 4,000 yds³/day	Process water no discharge (recycling) excess water- 0.2 ml/L SS 2,000 mg/L TSS (simple settling)	BCT=BPT	BAT=BCT=BPT	

POSSIBLE REVISIONS

Subcategory	BPT	BCT	BAT	NSPS
<1,500 yds/yr.	No limitations	None	None	None
>1,500 <70,000 yds³/yr. (small open-cut)	0.2 ml/L SS (Simple setting)	BCT=BPT	Process water- no discharge (recycling) excess water- 0.2 ml/L SS (simple settling)	NSPS=BAT
70,000-230,000 yds³/yr. (medium, open-cut)	0.2 ml/L SS (Simple settling	Process water- no discharge (recycling) excess water- 115 mg/L TSS (chemical treatment) or no limitation (simple settling)	Process water- no discharge (recycling) excess water- 0.2 ml/L SS (simple settling) or 0.2 ml/L SS and 115 mg/L TSS (chem. treatment)	same options as at BAT
>230,000 yds³/yr. (large, opencut)	0.2 ml/L SS (simple settling)	same options as for medium, open cut mines	same	same
Dredges	no discharge (recycling) excess water- 0.2 mg/L SS (simple settling)	same options as medium and large open-cut mines	same	same

APPENDIX B

Sample Solution Preparation Procedure for DCP Spectrometric Analysis

Mix 0.2 g of -200 mesh sample with 1.00g of lithium metaborate, transfer to a high-purity graphite fusion crucible and place in a muffle furnace at 1000°C for 10 minutes. With a pipette, transfer 100.0 ml of the 4% HNO_3 dissolving solution to a flat-bottomed 200 ml teflon or polypropylene beaker and add a teflon-covered stirring bar. Remove the crucible from the furnace, swirl to gather uncoalesced beads of molten material and pour the melt into the beaker. Cover to limit evaporation loss, and stir gently over a magnetic stirring unit without heating until solution is complete. Transfer the solution to a clean plastic bottle with a tightly fitting cap. Pipet 4.0 ml of 2000 ppm lithium solution and add 60.0 ml of the 10 ppm Ge solution by means of a pipette. The concentration of the sample for the determination of Al, Ca, Fe, K, Mg, Na, Mn, Na, Si and Ti is 125 ppm.

APPENDIX C

Procedure For Flocculation Testing A 250 ml sample of thoroughly mixed wastewater is poured into a 400 ml beaker with a one-inch magnetic mixing bar. The beaker is placed on a magnetic stirrer. The scale on the magnetic stirrer is adjusted to vary the mixing speed. For conditioning (gentle agitation), the mixing speed is set at 3 on the magnetic stirrer scale. The mixing speed is about 400-500 RPM when a one-inch vortex is present. Chemicals in solution are added from a burette in less than one minute. Various mixing times were tested. The settling time is determined and the residual turbidity is measured after 5 minutes of sedimentation.